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**TRANSPORT AND FATE OF NITROAROMATIC AND NITRAMINE EXPLOSIVES
IN SOILS FROM OPEN BURNING/OPEN DETONATION OPERATIONS:**

RADFORD ARMY AMMUNITION PLANT (RAAP)

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PREFACE

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**TRANSPORT AND FATE OF NITROAROMATIC AND NITRAMINE EXPLOSIVES
IN SOILS FROM OPEN BURNING/OPEN DETONATION OPERATIONS:**

RADFORD ARMY AMMUNITION PLANT (RAAD)

1.

INTRODUCTION

a. Out-of-date and out-of-specification munitions have commonly been disposed of by burning, or by detonation, on unprotected ground.¹ Through the promulgation of various environmental regulations, this practice has recently been limited. Burning pans and closed treatment systems have been used at various installations to mitigate environmental contamination. However, questions concerning the transport and transformation of open burning/open detonation (OB/OD) ash and waste explosives in soils and their environmental toxicity needed to be answered (AEHA, 1986).²

The standard practice of OB/OD of munitions historically involved quantities of explosives up to thirty tons per disposal event, and generated a mixture of contaminants into the immediate area at high concentration.³ At many military installations OB/OD sites consist of multiple disposal areas. These OB/OD sites number in the hundreds, and have been developed and used by both the military and their civilian contractors during much of this century. Many of these sites have records inadequate to predict the nature and extent of the contamination. Residue from OB/OD contains both burned and unburned explosives, but environmental weathering and microbial action are known to produce modifications of these compounds.^{4,5,6} Estimation of the environmental impact of OB/OD contamination at an individual site requires detailed knowledge of the type and amount of the chemical contaminants present and an understanding of their migration behavior within the soil.

The purpose of this project was to:

1) determine the transport and transformation of OB/OD contaminants in soil, 2) measure the toxicity of soils contaminated with explosives and 3) measure the toxicity of soil leachates. Three tasks were conducted to address the goals of the program. The first task used intact soil columns to measure the transport and transformation of chemicals in OB/OD ash and explosives of concern. The other two tasks involved determining the toxicity of explosives in soil to earthworms, and the toxicity of aqueous soil extracts to *Daphnia magna*.

In task one, intact soil cores were collected from Radford Army Ammunition Plant (RAAP), Virginia; Milan Army Ammunition Plant (MAAP), Tennessee; Pueblo Army Depot (PAD),

Colorado; and Anniston Army Depot (AAD), Alabama. The predominant explosives at each site were monitored in their respective soil-core columns for transport and transformation in the soil. Breakthrough and subsequent concentrations of the chemicals in the leachates collected from the columns were determined. Chemical transport and transformation experiments involved leaching soil columns with synthetic rainwater for up to 243 days. This report presents the data for Radford Army Ammunition Plant soils.

In task two, standard 14-day earthworm toxicity tests were conducted on OB/OD residues and specific explosives (results reported separately, in another technical report entitled **Toxicity of Selected Munitions and Munition-Contaminated Soil to the Earthworm *Eisenia foetida***).⁷ In task 3, soil/water extracts were prepared, to partition water soluble biologically available components from the soil. These aqueous extracts were tested for toxicity to the aquatic organism *D. magna* (results reported separately, in another technical report entitled **Determination of Soil Toxicity to *Daphnia magna* Using an Adapted Toxicity Characteristic Leaching Procedure**).⁸ The sensitivity of the *D. magna* method makes it a useful tool in assessing the impacts of contaminated soils. The results of this project will support site closure assessments at OB/OD sites, answer critical questions on the transport of explosives in soil, and address environmental toxicity data gaps.

In task one intact soil-core columns were collected on-site to study the transport and transformation of munition residues in site-specific soils. Intact soil-core columns were collected rather than collecting bulk samples of soil for packed-column studies because soil physical and chemical characteristics are typically, sometimes dramatically, altered by the drying, sieving, and storing of soils necessary for preparing packed columns. Furthermore, such handling may also cause inappropriate and radical change in the ability of soil to degrade xenobiotics⁹ or utilize naturally occurring compounds.¹⁰ Intact soil cores offer the potential for a realistic view of site-specific soil conditions as they exist in the field, yet are portable so they may be studied closely in the laboratory under conditions that simulate those occurring in the field. If appropriate precautions are taken during the collection, transport, and study of intact soil cores, information obtained for site-specific soil conditions may also give added insight to the processes controlling the transport and transformation of munition residues in soils. Many investigators acknowledge the advantages of using intact soil cores for study, but apply methods that require at least one transfer of the soil core from the collection probe to its destination column, potentially causing disruption of the soil core and alteration of its characteristics. However, a group of scientists^{11,12} have developed a system for taking intact soil cores, and have applied

the system to the extent that it was accepted as a standard method for soil microcosm research by the U.S. Environmental Protection Agency¹³ and the American Society for Testing and Materials.¹⁴ The system used during the investigations detailed in this report is an adaptation of those soil microcosm methods, with various refinements to more realistically assess the transport and transformation of chemicals in soils.¹⁵ The methods presented the following section (II. Soil Methodology) describe these improved methods for 1) taking and directly delivering soil cores into their respective columns with minimal disturbance of the soil sample; and for 2) controlling environmental parameters of the soil cores during study including soil temperature and moisture regime, including quantity, quality, and intensity of simulated rainfall. These factors directly impact on the chemical, physical, and biological properties of the soil, and potentially affect the resulting transport and degradation of chemicals within soil¹⁶ and their toxicity.¹⁷

RAAP was selected as the first site for collection of samples, characterization, and investigation. RAAP has an open burning area, and has burned waste explosives from their manufacturing operations containing 2,4,6-trinitrotoluene (TNT), 2,4-dinitrotoluene (2,4-DNT), and 2,6-dinitrotoluene (2,6-DNT). Burning operations were carried out in burning pans, however contamination of the soil did occur.

a. Collection of Intact Soil Cores

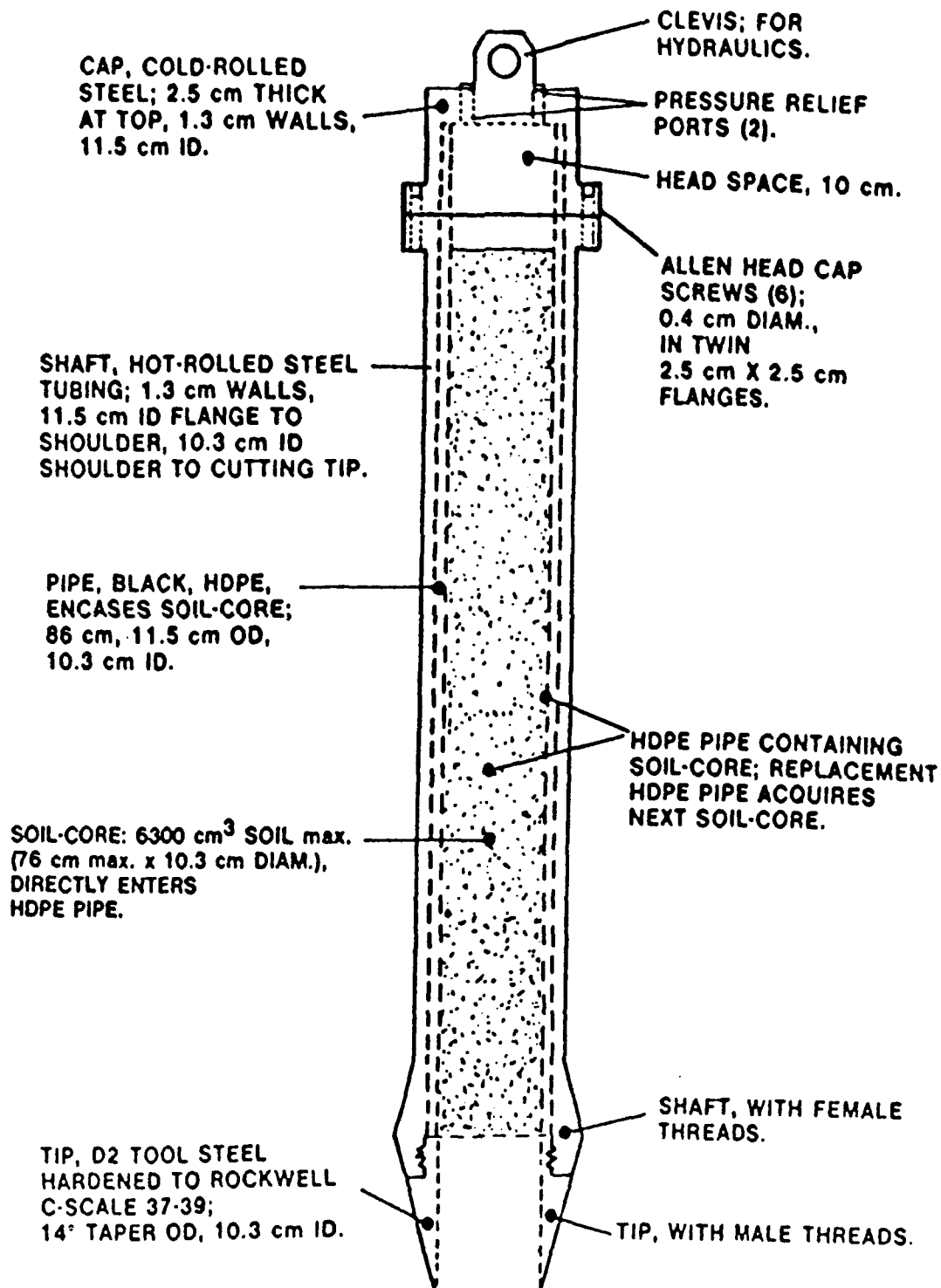
Prior to initiating collection of soil cores, a visual inspection of the OB/OD site was performed to ensure that the soil types conformed to those specified in the soil survey maps, obtained from the U.S. Soil Conservation Service.¹⁸ Next a site of the same soil type and characteristic as that of the OB/OD area was located. In order to be selected, a site must be free from contamination by munition residues, preferably undisturbed, and have an area large enough that sampling near soil-type transition areas or obvious physical discontinuities was avoided.

In the field prior to sampling on-site, the soil was brought to field moisture capacity. Watering of the soil was initiated at least 24h before sampling to ensure sufficient time for both wetting, and drainage of excess water. A sampling grid was then layed out at the site selected so soil-cores would be taken every 4 feet, on center. This was done to ensure that there was sufficient work area around each sampling location to prevent compaction of adjacent locations during sampling. Each site was measured and sampling locations were marked with flags. Native vegetation (primarily grasses) were cut at the soil surface and the aerial portions of the cut plants were removed prior to sampling the soil.

The probe (Fig. 2.1) was lifted into the air and moved to each sampling location using the front-end loader and a chain. An aluminum stop-plate, 18" x 18" x 0.5" (45 cm x 45 cm x 1.3 cm) with a central hole for locating the probe, was placed over the sampling location prior to pushing the probe into the soil. The stop-plate allowed more uniform samples to be taken. A total of thirty soil-core samples were taken per site to ensure an excess of available columns¹⁹ from which to initially test and ultimately select the final twelve columns per study. The soil probe was pushed rather than pounded into the soil to alleviate zonal compaction and minimize disruption of the soil being taken.²⁰ To prevent disturbance of the soil at adjacent sampling locations, the front-end loader was brought in perpendicular to the area in its approach to the first sampling location; after the sample was taken, the loader was backed out, moved to the right, again moved in perpendicular to the next sampling location; and this process continued until sufficient soil-core columns had been collected.

For the soil that entered the probe during collection of intact cores, the maximum clearance discrepancy allowed (using the tolerances specified, Fig. 2.1) during delivery of soil into the high density polyethylene (HDPE) pipe

FIGURE 2.1 CROSS-SECTION OF SOIL SAMPLING PROBE WITH SOIL-CORE ENCASED IN HDPE.



inside the probe was <0.05 -cm, resulting in a soil-core diameter of $10.3\text{-cm} \pm 0.1$. The HDPE pipe used in this study was opaque, the grade and quality used in high pressure gas pipelines. HDPE pipe was purchased in 12.2-m (40-ft) lengths, and prior to going to the field was cut and sanded to the specified dimensions. The HDPE pipe collection tubes were inert hydrophobic barriers that remained an integral part of the soil-core columns. Thus, disruption of the soil due to column-to-column transfers was eliminated. Upon removal of the HDPE collection tube containing the soil-core from the probe, measurements were taken of the resulting head space within each column; additionally it was advantageous to measure the depth of soil penetration by the probe that results from sampling. If dramatic inconsistencies occurred in the depth values in the field, the corresponding columns were rejected and others taken in their place. After removal from the probe, each HDPE collection tube containing a soil core was immediately placed in a set of "V" blocks for sealing and packaging. Each end of the HDPE collection tube was sealed with a barrier-cap consisting of double layers of 4-mil thick polyethylene sheeting, then sealed with duct tape to the HDPE pipe. This minimized gas exchange and prevented moisture loss from the soil cores. A sufficient supply of barrier-caps were prefabricated in the laboratory, prior to going to the sampling site, in order to decrease the amount of field time required to seal a soil-core sample tube. Barrier-caps were prefabricated by cutting out a 10" square piece of double-layered (2 x 4-mil) polyethylene sheeting, centering the square over an empty HDPE collection tube, and wrapping it around while pushing it down over the tube. This wrap was then held in place by a thick rubber band so a piece of duct tape could be placed tightly around the wrap 1" (2.5 cm) from the end of the HDPE collection tube. The corners of the square wrap (excess) were then cut off around the tube 2" (5.0 cm) below the tape. When using these barrier-caps in the field, the barrier-cap is slipped onto the end of the HDPE collection tube and an additional piece of duct tape is used to completely seal the edge of each barrier-cap to the outer surface of the tube. After the ends were sealed, each tube was labeled with the date, location, and collection site number.

Collected soil cores in their HDPE tubes were placed into 32-gal (120-L) opaque polyethylene containers, which contained a 6" (15 cm) thick foam rubber pad in the bottom. A group of HDPE tubes were placed on the pad in each container with the soil end down. The sealed columns extended out of the top of the containers, and through the container covers which had been cut to fit the columns. Black polyethylene plastic bags were used to cover the tops of the sealed columns. All soil samples obtained from a site were transported back to the laboratory upright in padded containers to minimize disruption of the soil cores during transport.

b. Soil Column Preparation and Testing

Afterward in the laboratory, selected soil-core columns were trimmed of excess soil if any was present, fitted with a porous ceramic disk (2.5 μm pores) in opaque HDPE endcaps containing fittings for teflon tubing with in-line monitoring and shut-off valves (Fig. 2.2). The HDPE end-caps used in this study were the grade and quality used in high pressure gas pipelines, however prior to use each was milled to contain a well for the controlled-pore ceramic plate, then milled again and threaded for tubing fittings. End-cap fittings were also HDPE. The in soil-core columns were then transferred into the controlled temperature (controlled environment soil-core microcosm unit; CESMU) chamber (Fig. 2.3). The CESMU chamber was housed in a greenhouse for high-temperature control, and was equipped with 10.5 MJ h⁻¹ cooling capacity sufficient for maintaining a constant temperature within entire soil columns for isothermic studies at 25.0 \pm 0.1 $^{\circ}\text{C}$. During these investigations the tops of the columns were left open to receive sunlight, sufficient for plant growth (however, they could instead be covered with an opaque insulated cover spanning all columns to eliminate photodegradation processes). Controlled tension (vacuum) was applied equally at the bottom of each soil column across the controlled-pore ceramic plate, at 30-35 kPa; tension was regulated and monitored.

The tension that was applied is comparable to that encountered in the field as a result of combined soil matric and gravitational forces; thus avoided were undue flooding, the buildup of a hanging column of water in the lower portion of columns, and artificial changes in soil redox potential in response to steady-state alteration of the soil water content, as can happen when gravitational forces alone are relied upon to promote water flow through soil columns. Before initiating any studies of the fate, migration, and degradation of munition residues, the soil-core columns in the CESMU chamber were saturated with water and equilibrated under tension (48h minimum), after which water thru-put was evaluated for each of the initially selected columns.

The initial selection of twelve columns per soil type (site) for preliminary testing was done on the basis of similarity of head space within columns, an easily obtained measurement that is the complement to column length. Using the sampling methods and measurements described above, a group of columns differing in length by only centimeters (Fig. 2.4) was obtained that provided a sufficient number of columns from which to select those for the preliminary testing of water flow (thru-put). Soil-core columns were initially selected on the basis of similarity of length; and replacement columns within each soil type group, if needed, were those with the next closest

FIGURE 2.2 SOIL-CORE COLUMN INCLUDING END-CAP AND FITTINGS.

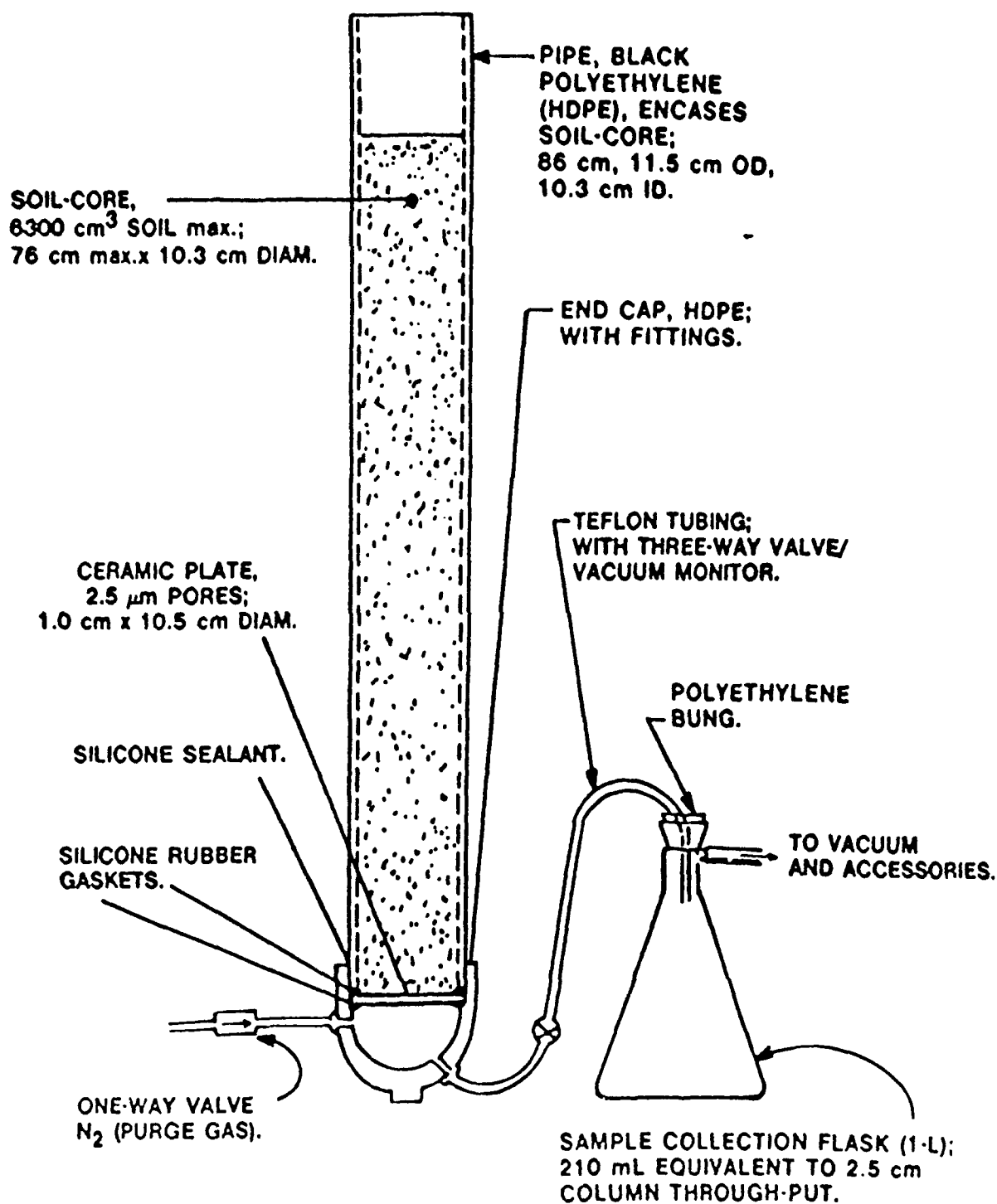


FIGURE 2.3 CROSS-SECTION OF CESMU SYSTEM SHOWING ONE SOIL-CORE COLUMN AND VACUUM SYSTEM.

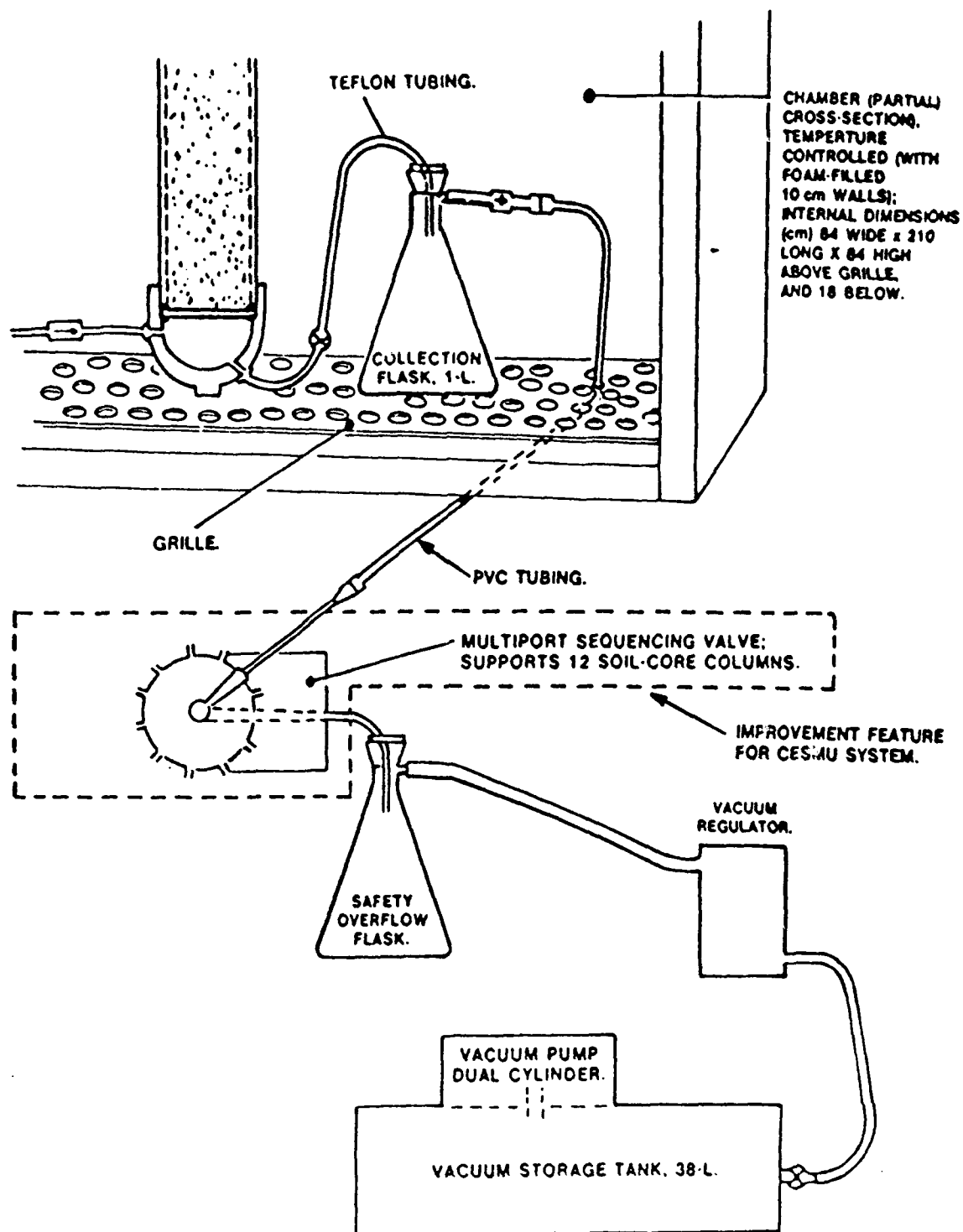
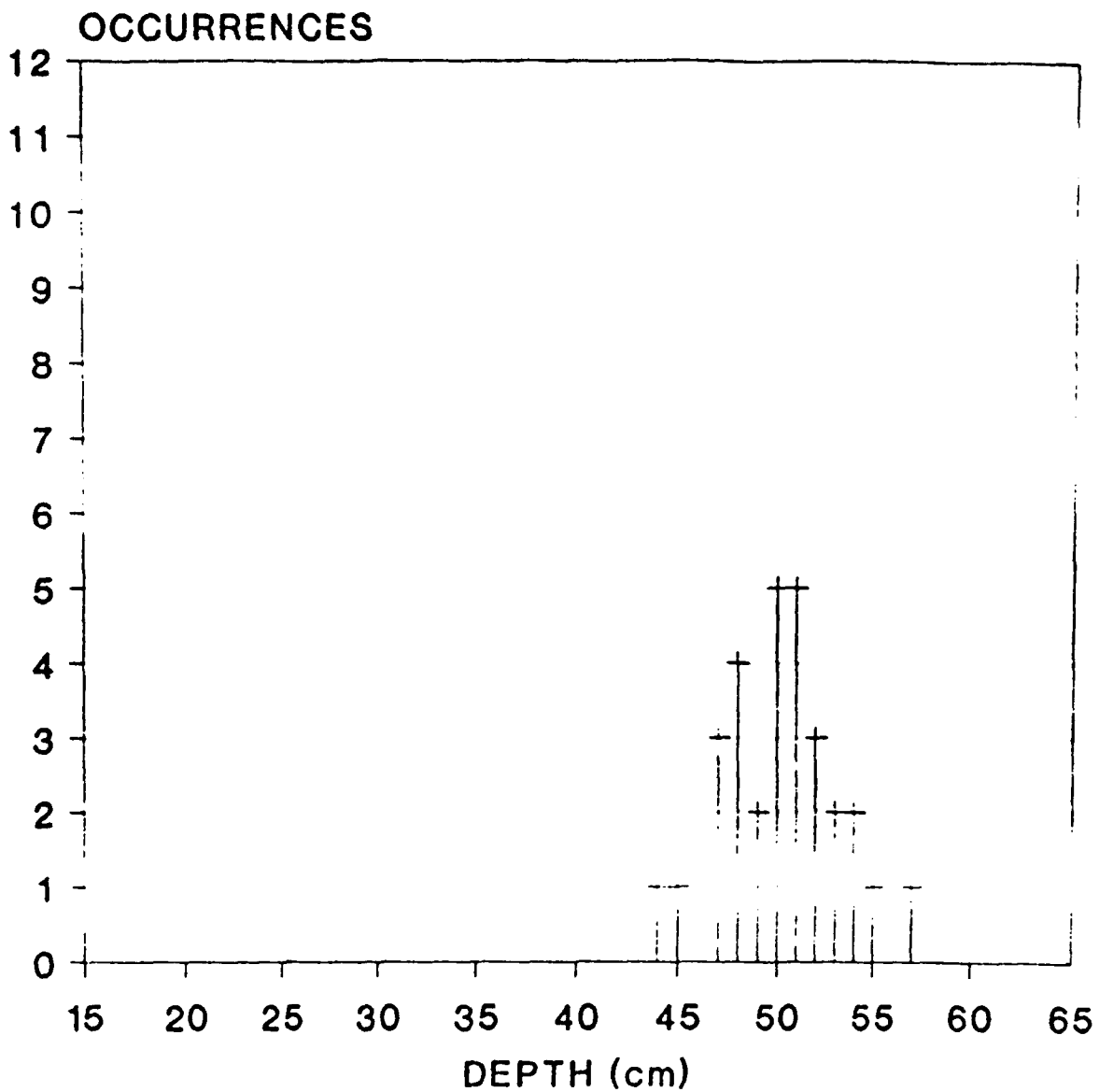


FIGURE 2.4 FREQUENCIES OF SOIL-CORE COLUMN DEPTHS: WHEELING SANDY LOAM SOIL (RAAP).



to the mean length. For the initially selected columns that were found to have rates of flow or water thru-put substantially different than the median, replacement columns were selected, and then similarly evaluated. Outlier-columns within each soil type (based on values of water thru-put, when water was applied, monitored, and sampled analogous to artificial rain additions described below) were replaced until the standard deviation about the mean value for water thru-put was $\leq 10\%$. Then, based on the adjusted mean excluding outliers, any additional columns with thru-put values falling outside of the adjusted mean \pm original standard deviation were also replaced, until all test columns fell within one standard deviation of the mean. Representative columns were thus identified and retained for study in the CESMU chamber.

c. Spiking of Soil Columns

OB/OD contaminated soil was collected from an open detonation pit that had the most recent disposal operation. This contaminated PAD soil was air-dried, extraneous materials (nails, stones, etc.) removed, crushed, and ground to pass a 2-mm nylon sieve. After this, the type and quantity of munition residues was determined. Then a mixture of the prepared detonation pit soil and explosives, related to munition residues detected in the screening analysis, was prepared.

Soil contaminated with residues from OB/OD activities was collected, and the type and quantity of munition residues determined. A mixture of the native soil and the type of munition residues detected in the screening analysis was prepared. After twelve representative soil columns collected from the site were identified and randomly placed in the CESMU according to the specifications in this report, the mixture of soil and explosives was added atop the soil surface of the randomly assigned treatment columns. Thus each third of the CESMU chamber contained a randomized complete treatment block, consisting of three treatment columns and a control column. During preparation of the mixture, 125.0 mg kg^{-1} 2,4-DNT and 40 mg kg^{-1} 2,6-DNT were added, while ambient concentrations of TNT and TNB were used. The spiking mixture was then analyzed and determined to contain the following concentrations of acetonitrile extractable explosives and transformation products (mg kg^{-1}): 61.7 ± 7.5 TNT, 103.8 ± 1.0 TNB, 115.8 ± 0.2 2,4-DNT and 36.6 ± 0.8 2,6-DNT. Each of the 9 treatment soil columns from the RAAP site received a mass of soil equivalent to 1" (2.5 cm) of A horizon soil (yielding approximately 210 mL of the mixture, after settling), while control columns received the same mass of uncontaminated soil from the site.

One inch (2.5 cm) synthetic rainwater (pH 4.60 ± 0.02 ; constituents detailed in this report was administered at the top to the center of each soil-core column

twice a week at the rate of $1'' \text{ h}^{-1}$ (7 um s^{-1}) using a peristaltic pump. Resulting leachates were collected into vacuum flasks and kept at soil column temperature (25.0°C). Leachates were harvested bi-weekly, and analyzed for munition residues and transformation products; the pH of leachates was determined at the time of collection. The maximum duration of leaching was 39 weeks.

Harvest of Soil Columns

Four soil-core columns (three treatment and one control) were harvested after each designated leaching interval. Harvesting of columns occurred after 13, 26, and 39 weeks of leaching, respectively. Column harvest, sectioning, and preparation for analyses, are described in this report.

d. Analytical Methods

The analytical methods and procedures for determining munition residue concentrations in leachates were the same as described in Section 3 of this report, with the following exceptions:

i. The extracts of soil sub-samples were filtered through a Gelman 0.45 um Acrodisc-CR disposable filter.

ii. Sub-samples of the aqueous leachates were adjusted to $\text{pH } 6.00 \pm 0.05$ and made to contain $300 \text{ g L}^{-1} \text{ NaCl}$. Two hundred mL of the resultant solution was put through a J.T. Baker 40 um Sep-Pak Octadecyl (C18) disposable cartridge at a rate of 1.8 mL min^{-1} . Cartridges were prepared for use by wetting with 2 mL methanol, followed by 2 mL deionized water. Munition residues were eluted from the cartridges with $2 \times 1\text{-mL}$ additions of methanol, and eluates were analyzed by HPLC.

Criteria of detection values for treatment soil samples for each explosive and their transformation products, including details of calculation, are given in Appendix B.

e. Simulated Rainfall and Resulting Leachates

In the laboratory, synthetic rainwater was formulated based on records of the constituents of rainfall across Pennsylvania,^{21,22,23} and used to represent the constituents and characteristics of rainfall in the mid-Atlantic coastal region. The constituents of the synthetic rainwater were (uM , in deionized water) 15 SO_4 , 11 NO_3 , 9 Cl , 25 NH_4 , 7 Ca , 3 Mg , 3 Na , and 2 K ; pH was adjusted to 4.60 ± 0.02 using a $1.35:1$ mixture of $1\text{M } \text{H}_2\text{SO}_4$ and $1\text{M } \text{HNO}_3$. Synthetic rain was administered at the top to the center of each soil-core column twice a week at the rate of $1'' \text{ h}^{-1}$ (7 um s^{-1}) using a peristaltic pump.

Resulting leachates were collected via teflon tubing into 1-L flasks in darkness, and kept at soil column temperature (25.0 ± 0.1 °C) inside CESMU until removed for analyses. Nitrogen gas was provided for purging the end-cap, but only during collection of the aqueous leachates. Leachates were harvested at regular intervals, and analyzed for munition residues and transformation products; the pH of leachates was determined upon collection using a combination pH electrode and digital pH multimeter.

Synthetic rainwater (pH 4.60 ± 0.02) in the amount of 0.2" (0.6 cm) was administered at the top to the center of each soil-core column twice a week at the rate of $1" h^{-1}$ ($7 \mu m s^{-1}$) using a peristaltic pump. Resulting leachates were collected into vacuum flasks and kept at soil column temperature (25.0 °C). Leachates were harvested twice-weekly, and analyzed for munition residues and transformation products; the pH of leachates was determined at the time of collection. The maximum duration of leaching was 32.5 weeks.

f. Harvest of Soil Columns

Replicate soil columns were harvested at regular intervals following leaching, sealed (in the same manner as when collected from the field, Section 2.b), then frozen. Afterward, the frozen soil cores encased in HDPE pipe were carefully cut open using a router (with the depth of penetration set to the wall thickness of the HDPE tubes) and a hand guide, allowing the resulting intact soil core to rest in the lower half of the HDPE pipe. Soil cores were then slowly thawed in the horizontal position to effectively eliminate longitudinal migration. Then from top to bottom, the soil cores were marked into sections using a spatula to indicate 1" (2.5 cm) depth intervals. The soil was then sectioned into 1" depth x 4" diam. (2.5 cm x 10.3 cm) discs. Each disc was individually transferred into a clean polyethylene bag, air-dried, crushed, and ground to silt consistency ($\leq 150 \mu m$). Using similar sectioning methods but larger section sizes, replicate bulk density determinations were done individually for A and B horizons using the extra soil-core columns.

g. CESMU System Integrity

Although controlled tension was applied equally at the bottom of each soil-core column during studies and was regulated and monitored, the failure to maintain tension at any single column potentially affected the tension on the remaining columns until the failing column was repaired or eliminated. Generally this problem occurred only during the set-up and preliminary testing of columns, and resulted from an immediately repairable minor leakage. Infrequently, this problem occurred due to handling of system components during sampling of leachates, but again caused only minor leakage of vacuum and was easily and

immediately repairable.

Physical and mechanical systems supporting the CESMU chamber and rainfall delivery functioned well under almost constant use for more than two years. Over this period, the transport and transformation of munition residues were investigated in four different site-specific soils, using twelve study columns per soil type (site), with individual studies lasting from six to nine months depending upon the lability of chemicals investigated. During these studies only one study-column failed out of forty-eight total columns selected for investigation, and the remaining soil columns had relatively constant outputs within respective soil types.

Mechanical-part failures during this period included only one vacuum pump failure (replaced with a back-up unit while the original was rebuilt), and one vacuum regulator that failed inspection during an investigation and was immediately replaced with a back-up unit. Performance of the physical and mechanical systems was high, providing high confidence in maintenance of the conditions and limits designed for the studies.

h. Determination of Selected Soil Parameters

For this investigation several soil physical and chemical parameters were selected for determination by the University of Maryland Soil and Plant Testing Laboratory, College Park, MD. The soil properties chosen were selected to more fully characterize and understand the role of the effects of specific soil properties on the transport and transformation of munition residues, and their transformation products. Soil properties determined included percent sand, silt, clay, and organic matter, the cation exchange capacity (CEC), and soil pH.

3. DETERMINING MUNITION RESIDUES AND THEIR TRANSFORMATION PRODUCTS

a. Analytical Methods Development Using High Performance Liquid Chromatography (HPLC)

The quality control program for this study was based on a system that assessed sample preparation, analyte recovery, and analytical precision and accuracy. Details of this program are presented in Appendix A.

Our approach to analytical determinations supporting these investigations was based on a two step process. The first step was qualitative analysis of contaminated surface samples to screen for compounds present in environmentally significant concentrations. Due to the variety of military explosives and their environmentally modified forms, a new method was required to chromatographically isolate and thus identify the majority of the compounds likely to be encountered. The second step was quantitation of these contaminants in soil and in water that leached through this soil. Screening and quantitation processes required different HPLC methods because quantitation required greater analytical sensitivity than the screening method could provide.

Sample preparation and extraction procedures were adapted from a method developed and extensively tested by Jenkins^{24,25,26}. These modified procedures entailed grinding air-dried soil samples, and extracting into acetonitrile with 18 hours of sonication at 20°C. Extracts were then centrifuged at 3900 X G for 15 min, and analyzed by HPLC. The latter portion of the sequence differs from Jenkin's method in that a step requiring mixing the acetonitrile extract with an aqueous flocculating solution was eliminated, and that the internal standard 1,3-dinitrobenzene (DNB) was incorporated.

An estimation of the efficiency of extraction of each compound was obtained by doping subsamples of uncontaminated surface soil with acetonitrile containing a mixture of selected OB/OD compounds plus DNB. The soil was air-dried and extracted as above, and the efficiency of extraction was calculated from the amount of each compound recovered. Because the efficiency of extraction of the OB/OD components at our test sites was similar to that of DNB, a simplified recovery correction system was possible. All soil samples were extracted with acetonitrile containing 2.5 mg L⁻¹ (ppm) of DNB as an internal standard. Observed concentrations of OB/OD components in the extraction mixture were corrected for losses of internal standard that occurred during the extraction process. Corrections were also made for any increases in concentration due to evaporation of the extraction solvent.

Aqueous leachates were directly analyzed for

munition residues and degradation products. These determinations were done without any preconcentration, internal standardization, or other preparation.

HPLC analyses of leachates and soil extracts were done using a Hewlett-Packard (HP) 1050 HPLC system that consisted of an autoinjector, pumping module, and UV detector. Signal integration was performed with an HP 3396A integrator. All analyses except screening tests for the presence of NG were done by UV absorbance at 244 nm. NG was determined at 220 nm.

Extracts of uncontaminated soils (background) and highly contaminated surface soils were screened by the gradient method developed for this investigation. A 15- μ L sample was injected onto a 4.6 X 250 mm Rainin Microsorb C18 column with a 5 μ m particle size, in series with a 4.6 X 250 mm Supelcosil LC-PAH column. Elution was accomplished with a methanol:water gradient (Table 3.1).

A simpler isocratic method (developed elsewhere by Miyares and Jenkins²⁷) was used to substantiate identification and to quantitate contaminants. This isocratic method entailed isocratic pumping of a mobile phase of 70.7% water, 27.8% methanol, and 1.5% tetrahydrofuran, at a flow rate of 2 mL min⁻¹

Table 3.1 HPLC Time/Gradient (Methanol:Water Mixture) for Initial Screening of Samples for a Broad Range of Munition-Related Analytes and PAHs.

| <u>Time (min)</u> | <u>Percent Methanol (% MeOH)</u> |
|-------------------|----------------------------------|
| 0 | 30 |
| 1.5 | 33.5 |
| 6.0 | 47.5 |
| 24.0 | 51.0 |
| 35.0 | 54.5 |
| 60.0 | 100.0 |
| 80.0 | 100.0 |

through a 25 cm x 4.6 mm Supelco LC8 column of 5 μ m particle

size. This procedure was modified by the addition of an acetonitrile gradient to minimize peak-broadening when amino-dinitrotoluenes (amino-DNTs) were quantitated.

b. Results of HPLC Methods Development

The above procedures have proven effective in recovering and quantitating OB/OD residues in all soils tested (Table 3.2); they have the additional advantage of being simple and reproducible. However, several shortcomings were encountered. Efforts to identify some minor components of the OB/OD soil contaminant mixture were not successful due to interferences from natural soil components. Although the majority of UV-absorbing soil components elute from reverse phase chromatography before most explosives, some elute at later retention times causing a rough baseline at high sensitivities thereby making quantitation of extremely small peaks unreliable.

Table 3.2 Efficiencies of Recovery of Selected Munitions, from Soil and Water.

| <u>Compound</u> | <u>Percent Munition Recovered (%), \pms</u> | | |
|-----------------|--|------------------|------------------------------|
| | <u>From soil extracted</u> | | <u>From aqueous leachate</u> |
| | <u>with acetonitrile</u> | <u>doped</u> | |
| | <u>doped</u> | <u>uncontam.</u> | <u>concentrates in MeOH</u> |
| RDX | 95 \pm 1 | 91 \pm 2 | 38 \pm 1 |
| HMX | 99 \pm 6 | 112 \pm 4 | 29 \pm 10 |
| TNT | 107 \pm 1 | 94 \pm 9 | 90 \pm 4 |
| 2,4-DNT | 103 \pm 1 | 110 \pm 5 | 108 \pm 7 |
| 2,6-DNT | 103 \pm 1 | 103 \pm 2 | 104 \pm 20 |
| 2-Amino-DNT | 100 \pm <1 | 103 \pm 1 | 112 \pm 15 |
| 4-Amino-DNT | 98 \pm 3 | 102 \pm 4 | 137 \pm 40 |
| TNB | 102 \pm 2 | 114 \pm 3 | 123 \pm 3 |

The gradient procedure presented here effectively separated components of a mixture that included most compounds likely to be encountered during analysis of soils from OB/OD contaminated sites (Fig. 3.1). It was able to detect many

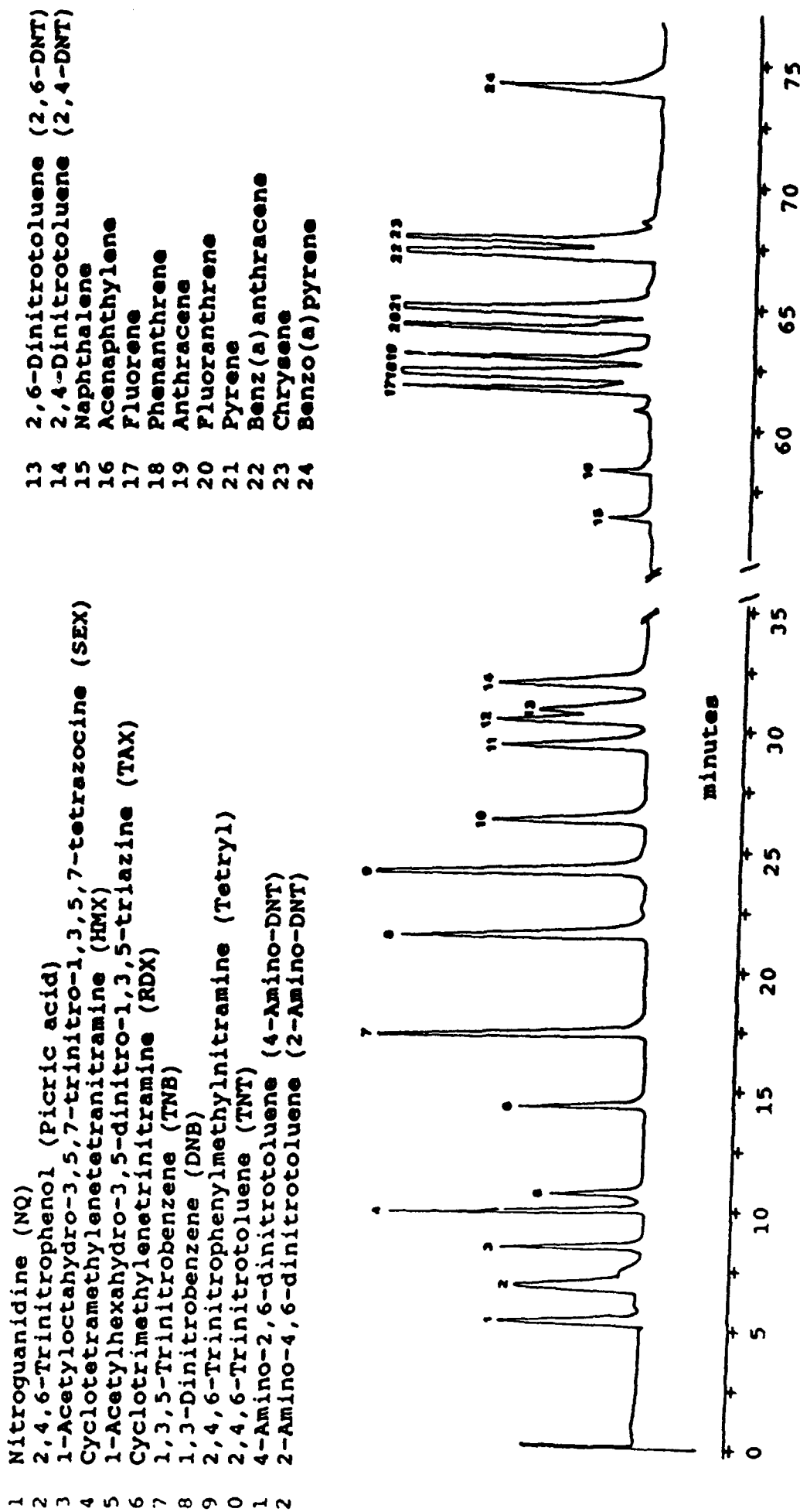


Figure 3.1 HPLC chromatogram showing the separation of a series of munition residues, explosives, and PAHs, using the gradient chromatographic (screening) method.

compounds that would otherwise be missed by previous methods, and produced sharp symmetrical elution peaks for all compounds tested. However this chromatography required 90 min to complete, and could not be used as a routine procedure at high sensitivity (compounds $<1 \text{ mg L}^{-1}$) because of problems with baseline drift. The isocratic HPLC method of Miyares and Jenkins proved effective in quantitating intact RDX, TNT, and DNTs (2,4-, and 2,6-dinitrotoluene) in water, acetonitrile, and methanol but performed less well with the aminodinitrotoluenes because they were later eluting and exhibited significant peak broadening (Fig. 3.2). Peak broadening caused problems with quantitation because it caused erratic start times during electronic integration of peak areas. We also observed that this solvent and column combination was unusually sensitive to temperature. At room temperatures the large negative absorbance peak from acetonitrile interfered with the quantitation of HMX. At temperatures above 23°C retention times were shortened, and at 30°C the system no longer resolved the two aminodinitrotoluenes.

Recovery of explosives doped into uncontaminated soil were nearly quantitative (Table 3.2); adjustments of recoveries due to gain or loss of the DNB internal standard were insignificant. Conversely, recoveries from the soil and water after leaching experiments ranged from 10-15% for TNT, 2-5% for 2,4-DNT, and even less for 2,6-DNT. Due to these low recoveries of the nitroaromatics from the leached soils, the concentrations of explosives in soil extracts, and in aqueous leachates, were often diminished to levels below our criteria of detection. The criterion of detection is defined as the lowest certifiable limit for quantitation. The respective criteria of detection were calculated using the computerized Quality Assurance Program of the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA),²⁸ based on the methods of Hubaux and Vos^{29,30}. Criteria of detection values were determined separately for leachate (aqueous) and soil samples for each explosive and transformation product, with details and calculations given in Appendix B. Criteria of detection for selected compounds are presented in Table 3.3, as a function of sample matrix.

When a compound was identified but quantitated to be at levels below the criteria of detection, it was termed to be a "trace" quantity and identified as **< criterion of detection**; a **zero value (0)** was reported when "no peak" was registered by the integration unit of the HPLC (i.e. not detectable) under the analytical conditions described in this report (above).

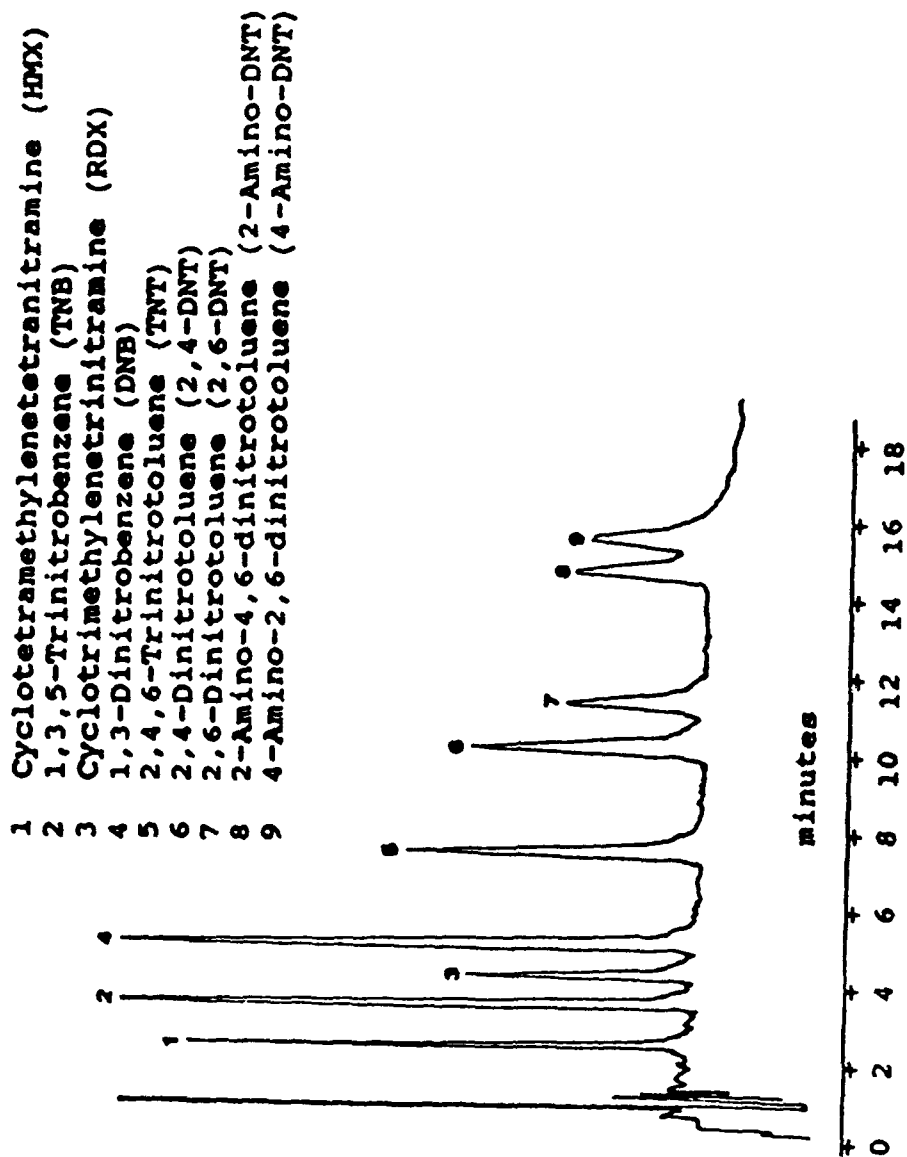


Figure 3.2 An example of the separation of a series of munition residues and associated co-contaminants, by the isocratic HPLC method¹².

Table 3.3 Criteria of Detection* for Selected Explosives and Their Transformation Products for Leachate (Aqueous) and Soil Samples.

| <u>Compound</u> | <u>Criteria of Detection by Sample Matrix</u> | |
|-----------------|---|--|
| | <u>Leachate</u>
<u>(mg L⁻¹)</u> | <u>Soil</u>
<u>(mg kg⁻¹)</u> |
| RDX | 0.07 | 5.8 |
| HMX | 0.14 | 2.9 |
| TNT | 0.09 | 6.1 |
| 2,4-DNT | 0.17 | 5.7 |
| 2,6-DNT | 0.37 | 5.2 |
| 2-Amino-DNT | 0.14 | 15.4 |
| 4-Amino-DNT | 0.12 | 14.6 |
| TNB | 0.15 | 2.4 |

* Calculations detailed in Appendix B.

c. Analytical Methods for Metals Determinations by Atomic Absorption Spectrophotometry

Concentrations of Cd, Cr, Cu, Pb, and Zn in uncontaminated soils and OB/OD contaminated ash/soil mixtures from each of the four OB/OD sites were determined in order to compare the background levels of metals in the respective soils with those of the contaminated/fortified (spiked) samples. Complete results from these analyses are reported in Appendix C. Duplicate 4.00 ± 0.02 g air-dried subsamples from each of the uncontaminated, contaminated, and contaminated/fortified (spiked) soils were each heated for 3 h on a hot plate in 20 mL 1.0 M trace-metal grade HNO₃. When the samples were cool, each was filtered by gravity through Whatman #50 paper, then brought to 50-mL volume with ultrapure water (reverse osmosis followed by double-deionization). All samples were analyzed for total extractable Cd, Cr, Cu, Pb, and Zn levels by atomic absorption spectrophotometry (Perkin-Elmer Model 3030 AA Spectrometer).

Quality assurance and control (QA/QC) for the metal determinations were achieved as follows. Absorbance and

concentration values for standard solutions were initially assessed to assure compliance with the values listed in the Perkin-Elmer methods guide. Standard solutions of the metals were periodically reread (absorbance redetermined) throughout the analyses for each metal determined, to check for instrument drift. Blank solutions were analyzed to detect any possible metal contamination. Additional subsamples were selected at random and prepared in replicate, to verify the analytical results obtained in initial analyses.

a. Results

i. Soil Parameters

The soil type at the RAAP OB/OD area consisted of Wheeling sandy loam soil (Fine-loamy, mixed, mesic, ultic, Hapludalfs),¹⁸ thus soil of this type was sought in an uncontaminated area on-site. Physical and chemical analyses of soil from the uncontaminated site confirmed the Wheeling sandy loam soil type. These soil parameter results are presented in Table 4.1.

Table 4.1. Physical and Chemical Characteristics* of Wheeling Sandy Loam from the Uncontaminated RAAP Site.

| | SURFACE
A HORIZON
(0-38 cm)
<u>0-15 INCHES</u> | SUB-SURFACE
B HORIZON
(38-61 cm)
<u>15-24 INCHES</u> |
|------------------------------|---|---|
| SAND % | 70 | 70 |
| SILT % | 22 | 22 |
| CLAY % | 8 | 8 |
| ORGANIC
MATTER
g/kg | 20 | 17 |
| CEC
cmol _c /kg | 8.2 | 9.7 |
| pH | 6.1 | 6.8 |

Values represent replicate determinations by the University of Maryland Soil and Plant Testing Laboratory, College Park, MD. *

Concentrations of all metals studied were higher in the contaminated than the uncontaminated Wheeling sandy loam soil (Appendix C). The concentration of each metal in contaminated soil was divided by the concentration in uncontaminated soil to reveal the anthropogenic elevation, in percent. Thus, relative concentrations of metals in contaminated soil were expressed as percentages of the values from uncontaminated background soil, followed by the determined concentration

values (mg kg^{-1}) for the contaminated soil: Cd 200% (1.1), Cr 160% (10.9), Cu 500% (30.0), Pb 1400% (183) and Zn 450% (303). On the basis of the anthropogenic elevations alone, the greatest potential environmental hazard from metallic residues at RAAP appears to be due to the elevated Pb concentrations in OB/OD contaminated soil.

Twelve uncontaminated Wheeling sandy loam soil columns having soil-core depths that were the most similar to the median were initially selected for preliminary evaluation in accordance with the procedures described in this report. Ten of these met the thru-put criteria while two did not. After replacing these two columns and testing the replacement columns, the set of twelve selected for spiking with contaminated RAAP soil and further investigation was successfully identified.

ii. Leachates

The volumes of leachates collected are given as a function of time in Appendix D, Table D-1. Concentrations of munition residues in RAAP soil-core leachates were determined by HPLC methods described in Volume I, Sections 2.d, 3, and 4.d (above). There were no detectable concentrations of munition residues or transformation products in any of the leachates harvested from RAAP Wheeling sandy loam soils, whether from control soil-core columns or soils contaminated with explosives (Appendix D, Tables D-2 and D-3).

The average pH values for each leachate harvest are given in Table 4.2. Generally, the pH of leachates tended to be quite high, and slowly decreased as leaching of the soil-core columns progressed. However, the average pH of leachates differed by less than one pH unit over the course of 274 days, during which the soil-core columns received 2" (5 cm) per week of synthetic rain ($\text{pH } 4.60 \pm 0.02$). Since the pH of the native Wheeling sandy loam was 6.1 in the A horizon and increased to only 6.8 in the B horizon, the higher pH of the leachates from the treatment columns may in part be due to solubilization of munition ash materials, and the action of soil microorganisms, both in the soil and in the resulting leachates which become inoculated naturally.

iii. Soil

Concentrations of munition residues in RAAP soils were determined by the HPLC methods described in this report. Results of analyses for each soil-core section, from all RAAP treatment and control soil-core columns, are given in Appendix D, Tables D-4.1 through D-4.12. The results from triplicate treatment soil-core columns are summarized in Table 4.3. The munition residues and transformation products that were

present in treatment soil-core columns at commencement of column leaching included TNT, TNB, 2,4-DNT, and 2,6-DNT. During this investigation of the transport and transformation of munition residues, the related compounds found in treatment soil-core columns included TNT, TNB, 2,4-DNT, 2,6-DNT, 2-amino-DNT and 4-amino-DNT.

Table 4.2 Average Leachate pH Values at Each Leachate Harvest Day for RAAP Soil-Core Columns that Received 1" (2.5 cm) Synthetic Rain (pH 4.60 \pm 0.02) Twice per Week for Up to 39 Weeks.

| Days | 12 columns | Average pH value | Standard Deviation |
|------|------------|------------------|--------------------|
| 14 | | 8.1 | 0.3 |
| 28 | | 7.8 | 0.2 |
| 42 | | 7.9 | 0.3 |
| 56 | | 7.9 | 0.3 |
| 70 | | 8.0 | 0.4 |
| 84 | | 7.9 | 0.4 |
| | 8 columns | | |
| 98 | | 7.9 | 0.4 |
| 112 | | 7.8 | 0.4 |
| 127 | | 7.8 | 0.4 |
| 144 | | 7.8 | 0.3 |
| 155 | | 7.8 | 0.3 |
| 168 | | 7.6 | 0.3 |
| 183 | | 7.6 | 0.4 |
| | 4 columns | | |
| 196 | | 7.7 | 0.4 |
| 210 | | 7.6 | 0.4 |
| 225 | | 7.6 | 0.5 |
| 239 | | 7.4 | 0.2 |
| 253 | | 7.8 | 0.4 |
| 267 | | 8.2 | 0.3 |
| 274 | | 7.8 | 0.3 |

b. Discussion

When the leaching of the soil-cores commenced, TNT, TNB, 2,4-DNT, and 2,6-DNT all were initially present in the top inch of soil of treatment columns (Table 4.3). TNT and TNB in the top inch of soil were extractable at 62 and 104 mg kg⁻¹ (ppm) respectively. TNB, a transformation product of TNT that is exposed on the surface at OB/OD sites, is frequently found in surface soils at concentrations exceeding that of the parent compound,³¹ and this was the case for the contaminated RAAP soil. When leaching commenced, no 2-amino-DNT or 4-amino-DNT (amino-DNTs) was present in the RAAP soil at detectable concentrations

even though in many environments TNT is microbially transformed by reduction to amino-DNTs.³² The 2,4- and 2,6-DNTs were added uniformly in the top inch of RAAP soil at the concentrations of 125 mg kg⁻¹ 2,4-DNT and 40 mg kg⁻¹ 2,6-DNT, resulting in initial extractable concentrations in soil of 116 and 37 mg kg⁻¹ respectively.

TNT, TNB, 2,4-DNT, and 2,6-DNT were transported vertically into the soil column, but to a depth of no more than three inches; as were 2-amino-DNT and 4-amino-DNT, unless created at depth within the RAAP treatment soils. After three months of leaching, the concentrations of extractable TNT, TNB, 2,4-DNT, and 2,6-DNT in the top inch (0-2.5 cm) of soil of treatment columns had declined by transformation of TNT *in situ* following transport. All of these munition residues migrated but to a very small degree, and resulted in very low concentrations only in the top 3" (0-7.5 cm) to 7, <1.3, <1.8, and <3.5 mg kg⁻¹ respectively, and remained at these levels through six additional (nine total) months of leaching; while concentrations of these compounds in the second (2.5-5 cm) and third (5-7.5 cm) inches of soil were approximately the same or even lower. TNT and 2,6-DNT concentrations in the top inch of soil declined by a factor of ten (one order of magnitude) or more, while TNB and 2,4-DNT concentrations decreased by a factor of 100 (two orders of magnitude) or more. Only trace amounts of the amino-DNTs (primarily 2-amino-DNT) were found in the leached RAAP soil, and these amounts were formed *in situ* (since none was originally present). The amino-DNTs are substantially more polar compounds than their precursor TNT, and such increased polarity generally leads to increase water solubility and thus mobility. The amino group may also destabilize chemisorption, allowing amino-DNTs to undergo ion exchange reactions at negatively charged soil surfaces. Both of these characteristics would tend to favor migration of amino-DNTs. However, even though more polar than TNT, the amino-DNTs were also found only in the top three inches of the RAAP treatment soils and only in trace amounts.

The dramatic decline in the concentrations of the munition residues in the RAAP soil did not cause high concentrations of any of these compounds in RAAP leachates. Not even trace concentrations of any of the compounds (or any other transformation products) were found in any of the leachates from the contaminated RAAP soil cores. Thus in this Wheeling sandy loam soil, the rate that TNT was transformed to either form of amino-DNT occurred at a very slow rate, as evidenced by their low concentrations in the soil, lack of presence in leachates, and lack of additional transport over time.

TNT, TNB, 2,4-DNT, and 2,6-DNT all became bound within the A horizon of the RAAP soil, in response to the simulation of natural weathering processes (i.e. alternating wetting and drying cycles, with the surface of the soil exposed to sunlight). The time-dependent disappearance of these munition residues in the environment may very well be due to covalent or other non-equilibrium

Table 4.3 Average \pm s Concentrations (mg kg^{-1}) of Acetonitrile Extractable TNT, TNB, 2,4-DNT, 2,6-DNT, 2-amino-DNT and 4-amino-DNT in 1" (2.5 cm) Sections of Triplicate Treatment Soil-Core Columns.

TIME ZERO (NO LEACHING)

| DEPTH | TNT | TNB | 2,4-DNT | 2,6-DNT | 2-amino-DNT | 4-amino-DNT |
|-------|---------------------|----------------|--------------------|-------------------|-------------|-------------|
| | mg kg^{-1} | | | | | |
| 1" | 62
± 7 | 104
± 1 | 115.8
± 0.2 | 36.6
± 0.8 | 0
- | 0
- |

Below this depth: No detectable concentrations (0).

THREE MONTHS OF LEACHING

| | | | | | | |
|------|--------------|-----------|-----------|-----------|-----------|-----------|
| 0-1" | 7
± 1 | <1.3
- | <1.8
- | <3.5
- | <1.4
- | 0
- |
| 1-2" | 2
± 1 | <1.3
- | <1.8
- | 0
- | <1.4
- | <1.0
- |
| 2-3" | 0
- | 0
- | <1.8
- | 0
- | <1.4
- | 0
- |

Below this depth: No detectable concentrations (0).

SIX MONTHS OF LEACHING

| | | | | | | |
|------|--------------|-----------|-----------|--------|--------|--------|
| 0-1" | 6
± 1 | <1.3
- | <1.8
- | 0
- | 0
- | 0
- |
| 1-2" | <1.3
- | <1.3
- | <1.8
- | 0
- | 0
- | 0
- |
| 2-3" | 0
- | 0
- | <1.8
- | 0
- | 0
- | 0
- |

Below this depth: No detectable concentrations (0).

NINE MONTHS OF LEACHING

| | | | | | | |
|------|--------------|-----------|------------------|-----------|-----------|--------|
| 0-1" | 6
± 3 | <1.3
- | 2.5
± 1.6 | <3.5
- | <1.4
- | 0
- |
| 1-2" | 3
± 1 | <1.3
- | <1.8
- | <3.5
- | <1.4
- | 0
- |
| 2-3" | 0
- | 0
- | 0
- | 0
- | <1.4
- | 0
- |

Below this depth: No detectable concentrations (0).

bonding to natural soil components. The A horizon of mineral soils, such as that at RAAP, usually reflect the influence of accumulated organic matter. Organic matter in soil tends to bind TNT and related compounds quite strongly and in very substantial amounts³³ especially if the binding capacity of the soil for these compounds has not been exceeded. Furthermore, though the aromatic ring structure of these compounds is resistant to degradation, evidence exists of other additional environmental processes in which these compounds may also become strongly bound to soil.

The concentrations of these munitions in RAAP soils were determined by sonically extracting the soil with acetonitrile, an efficient extractant for TNT, 2,4-DNT and 2,6-DNT and related compounds. Thus any chemically related munition residues not extracted from soil by sonication with acetonitrile are so strongly bound that they are effectively unleachable.

CONCLUSIONS*** Intact Soil Column System: CESMU**

A state-of-the-art controlled environment soil-core microcosm unit (CESMU) system was developed to determine the transport and transformation of chemicals in RAAP soil. The system used intact soil-core columns from the RAAP OB/OD site. The soil cores were put into the CESMU, where experiments were conducted under controlled environmental conditions. The major improvement of the CESMU system over existing microcosm technology was incorporation of a controlled weak vacuum to cause a continuous tension on the soil-core columns. This tension mimicked the effects of soil matric and gravitational tension in real world systems, allowing study of chemical transport and transformation under laboratory conditions.

*** Explosives and Transformation Products in Leachates and Soil**

There were no detectable concentrations of munition residues or transformation products in any of the leachates harvested from RAAP Wheeling sandy loam soils.

During this investigation of the transport and transformation of munition residues, the compounds found in treatment soil-core columns included TNT, TNB, 2,4-DNT, 2,6-DNT, 2-amino-DNT and 4-amino-DNT. All of these were transported vertically into the soil column, but to a depth of no more than three inches. TNT, TNB, 2,4-DNT, and 2,6-DNT all became bound within the A horizon of the RAAP soil, in response to the simulation of natural weathering processes (i.e. alternating wetting and drying cycles, with the surface of the soil exposed to sunlight).

*** Anthropogenic Elevation of Metal Levels in Soil**

Concentrations of all metals studied were higher in the contaminated than the uncontaminated Wheeling sandy loam soil. Relative concentrations of metals in contaminated soil expressed as percentages of the values from uncontaminated background soil, and determined concentration values (mg kg^{-1}) for the contaminated soil, were: Cd 200% (1.1), Cr 160% (10.9), Cu 500% (30.0), Zn 450% (303), and Pb 1400% (183). On the basis of the anthropogenic elevations alone, the greatest potential environmental hazard from metallic residues at RAAP appears to be due to the elevated Pb concentrations in OB/OD contaminated soil.

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APPENDIX A

QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

a. Analytical chemistry.

I. Analytical standards of explosives and related compounds were prepared by purification of existing USABRDL standards. Purification was accomplished by recrystallization in a water acetone system. A mixture of HMX, TNB, RDX, TNT, 2,6DNT, 2,4DNT, 2-Amino DNT, and 4-Amino DNT was prepared from analytical standards with each component at 100 ppm in acetonitrile. This mixture was sealed and stored at 2 to 5 degrees centigrade and used until expended (about six weeks).

II. The mixture was serially diluted with water or acetonitrile in a ten step process to yield calibration standards of 10, 5, 2.5, 1.25, 0.63, 0.32, 0.16, 0.08, 0.04, and 0.02 ppm. The standards were analyzed, peak areas recorded and a plot of concentrations/peak areas produced. Linear regression of this data in the form of $Y = MX + B$ with concentration as the dependent variable were calculated. This equation was used to calculate unknown concentrations from analyzed peak areas. New calibration standards were analyzed with each set of analytes run and the calibration curve recalculated.

III. Control samples to be analyzed with the test samples were prepared by diluting the multipart standard to 2.5 ppm with acetonitrile. Control samples were prepared in triplicate and analyzed with each batch of samples. The mean and standard deviation of these analyses were calculated and results from each analytical run plotted as scattergrams (Figures A1 to A9).

b. Extracts.

I. Soil columns were sectioned and soils ground and extracted in accordance with SOP and all extracts analyzed in triplicate. Quality assurance procedures were established to ascertain the efficiency of the extraction process. Uncontaminated soil samples were spiked after grinding with a mixture of the compounds under study and a percent recovery performed for each site (Table A1). Spiked samples were prepared in triplicate and analyzed with each batch of 27 soil extracts.

II. Dinitrobenzene (DNB) was added to the acetonitrile soil extraction solution as a means to provide an internal recovery standard for each soil sample analyzed. Separate samples containing only DNB and acetonitrile were analyzed in triplicate with each batch of soil extracts. Mean recovery and standard deviation of these samples were calculated as a check on extraction losses and analytical imprecision. These results are presented in Figure A10.

c. Leachates.

Aqueous leachates were collected within the CESMU and removed for analysis. Samples were then refrigerated until analyzed. Leachates were not concentrated and recoveries were not corrected by internal standardization.

d. Measuring devices.

Soils and explosives were weighed on scales of certified accuracy. Pipets were checked for accuracy when placed in service. Volumetric glassware was of certified accuracy.

e. Quality Assurance Categories for Investigation.

This investigation was initiated prior to the Toxicology Division SOP MGT-1 of 1 Oct. 91. However, this work meets the criteria of "Exploratory Research" in nature and is therefore classified as a Category 1 investigation. Good Laboratory Practices as applicable to this category of investigation, which were in place at the onset of work (Jan 1989), were followed throughout.

Fig. A1
HMX VARIABILITY IN THE MTPS

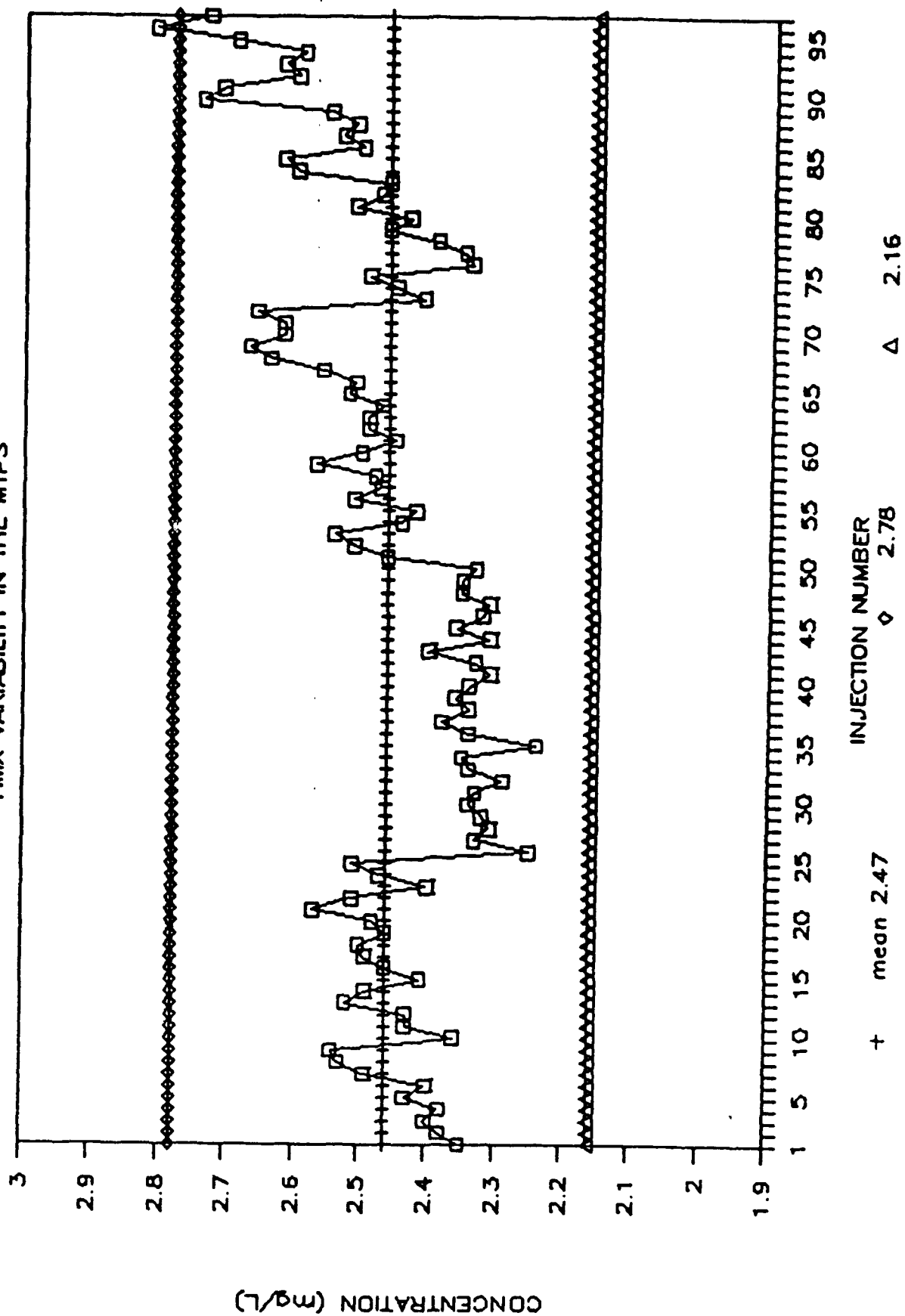


Fig. A2
TNB VARIABILITY IN THE MTPS

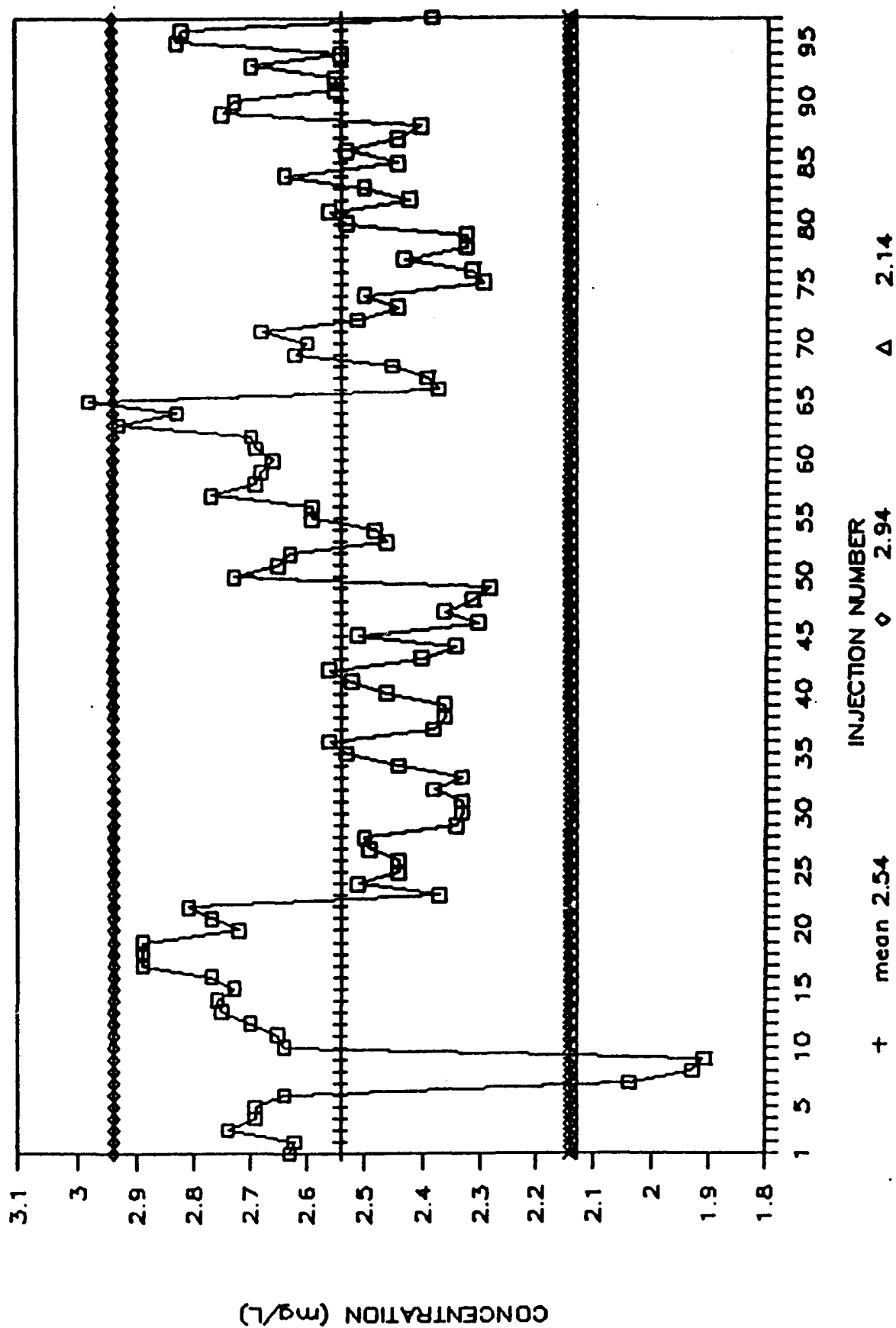


Fig. A3
RDX VARIABILITY IN THE MTPS

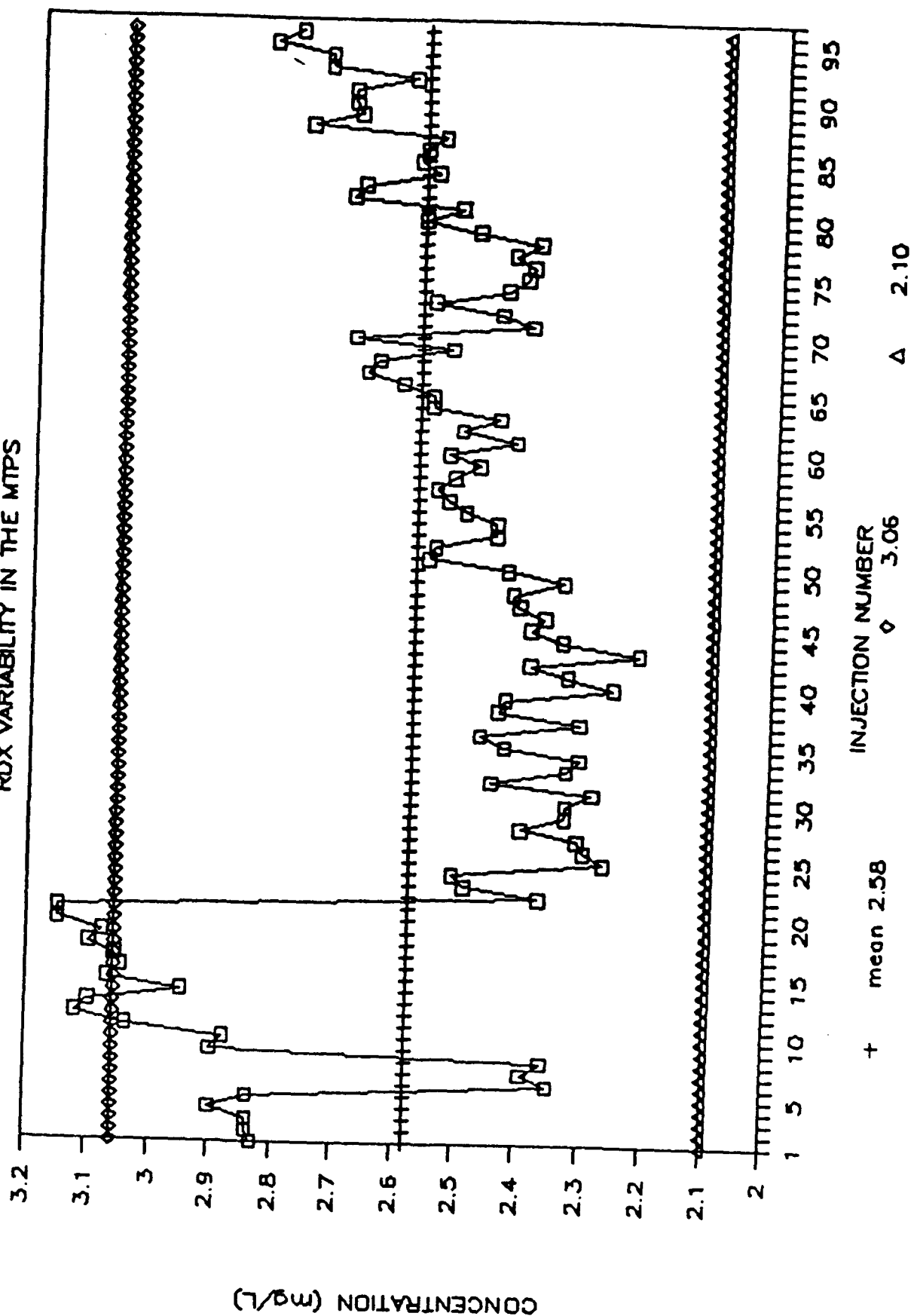


Fig. A4
TNT VARIABILITY IN THE MTPS

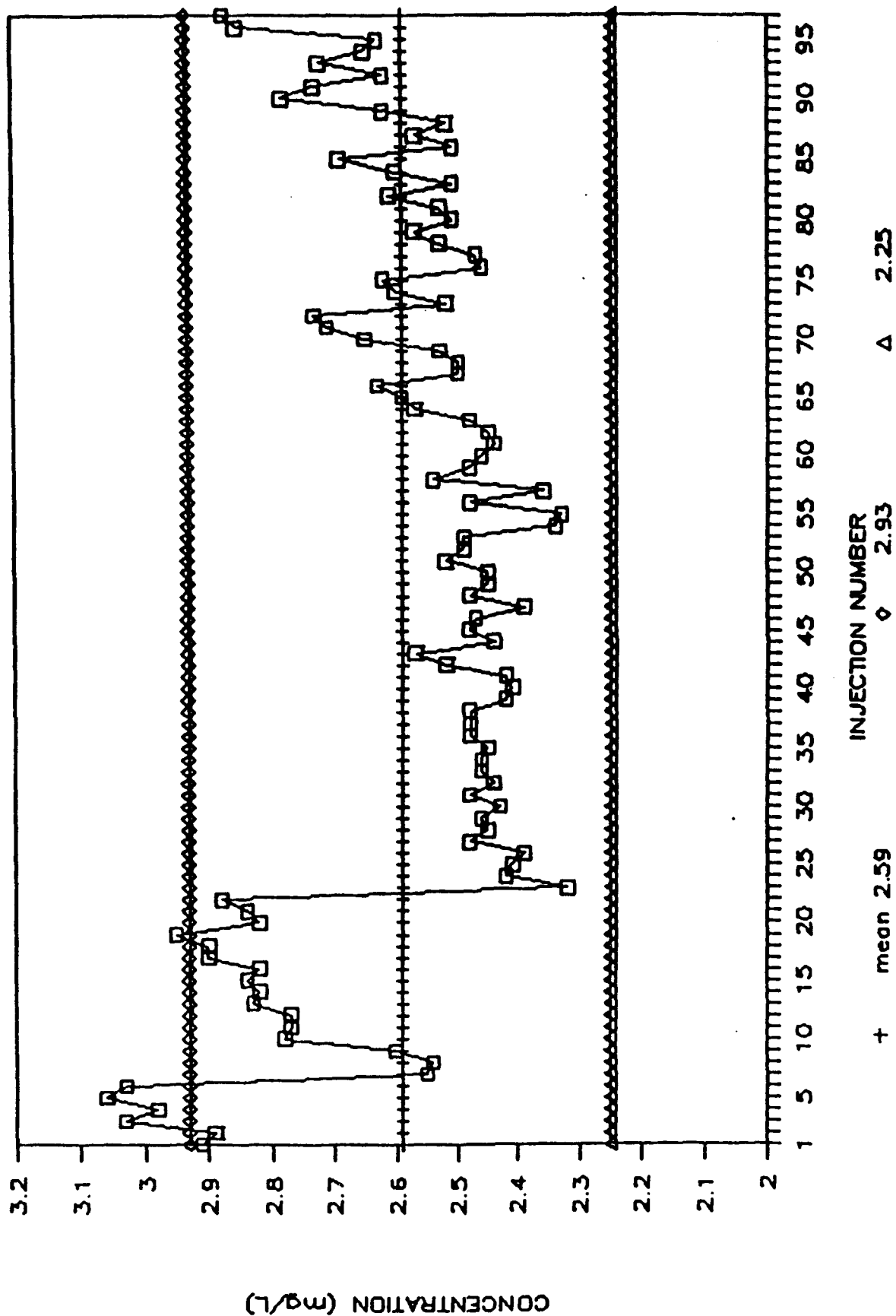


Fig. A5

2.4 ONT VARIABILITY IN THE MTPS

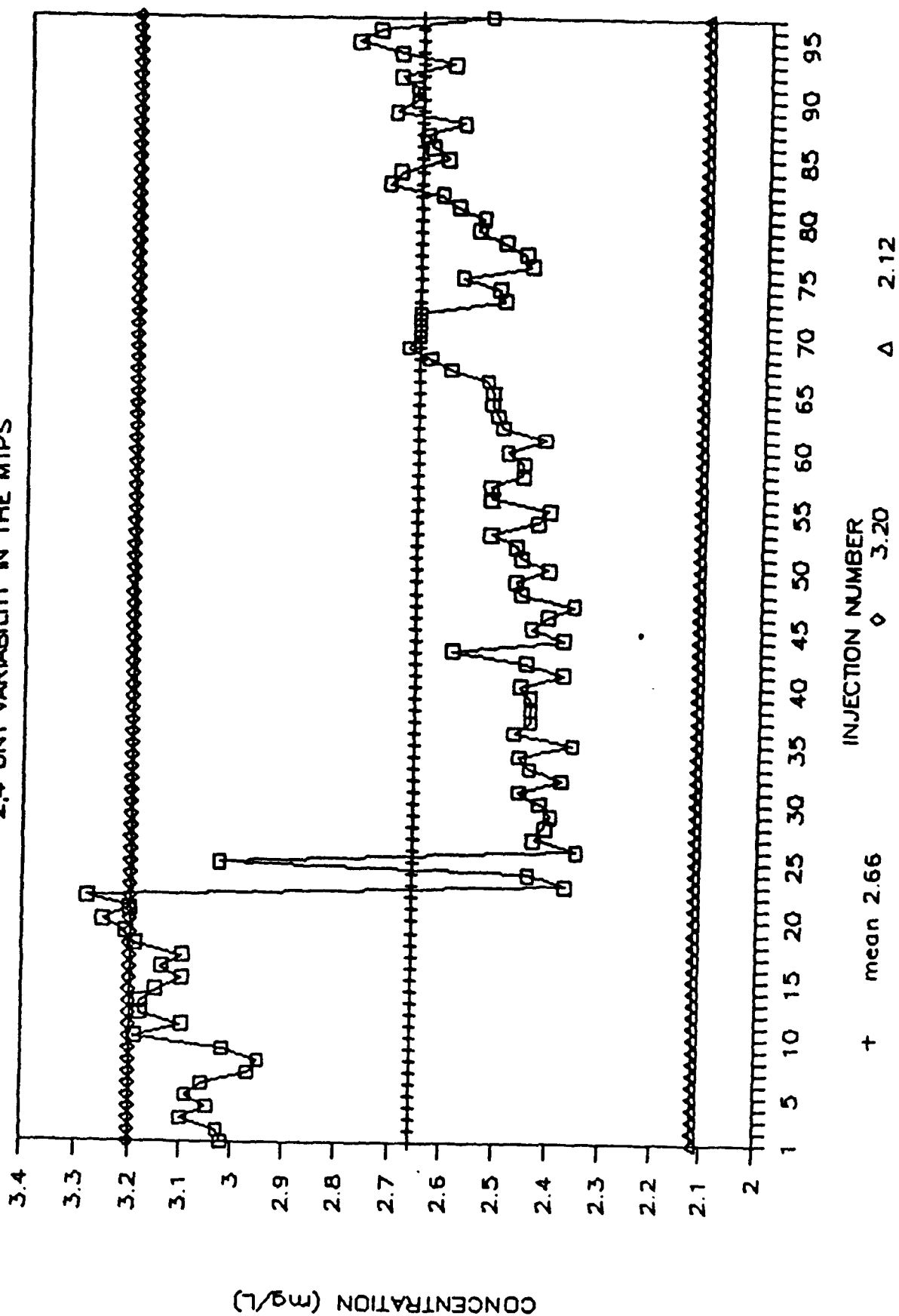
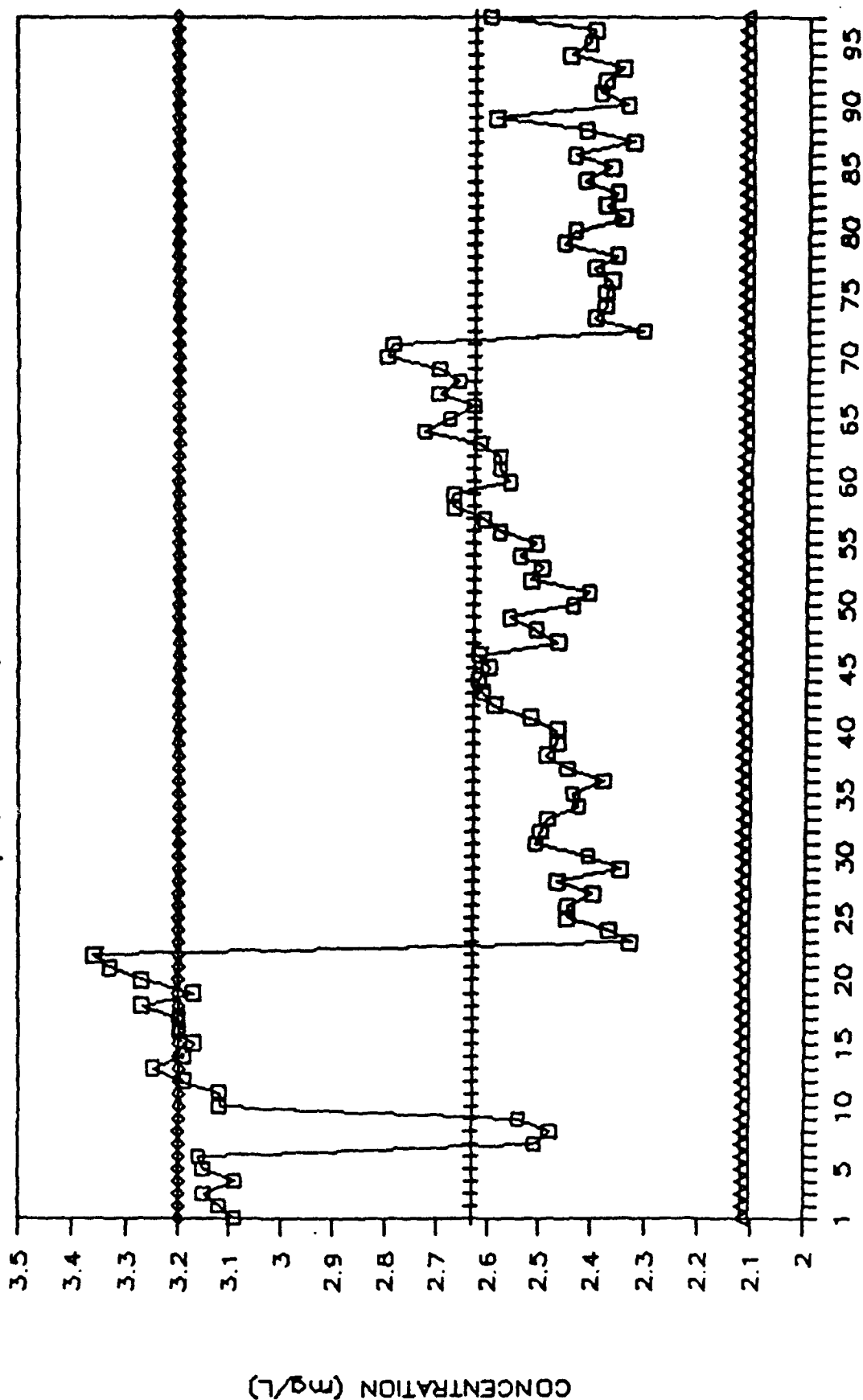


Fig. A6

2.6 ONT VARIABILITY IN THE MTPS



+ mean 2.63 INJECTION NUMBER Δ 2.12

Fig. A7
2-AMINO 4,6 DNT VARIABILITY IN THE MTPS

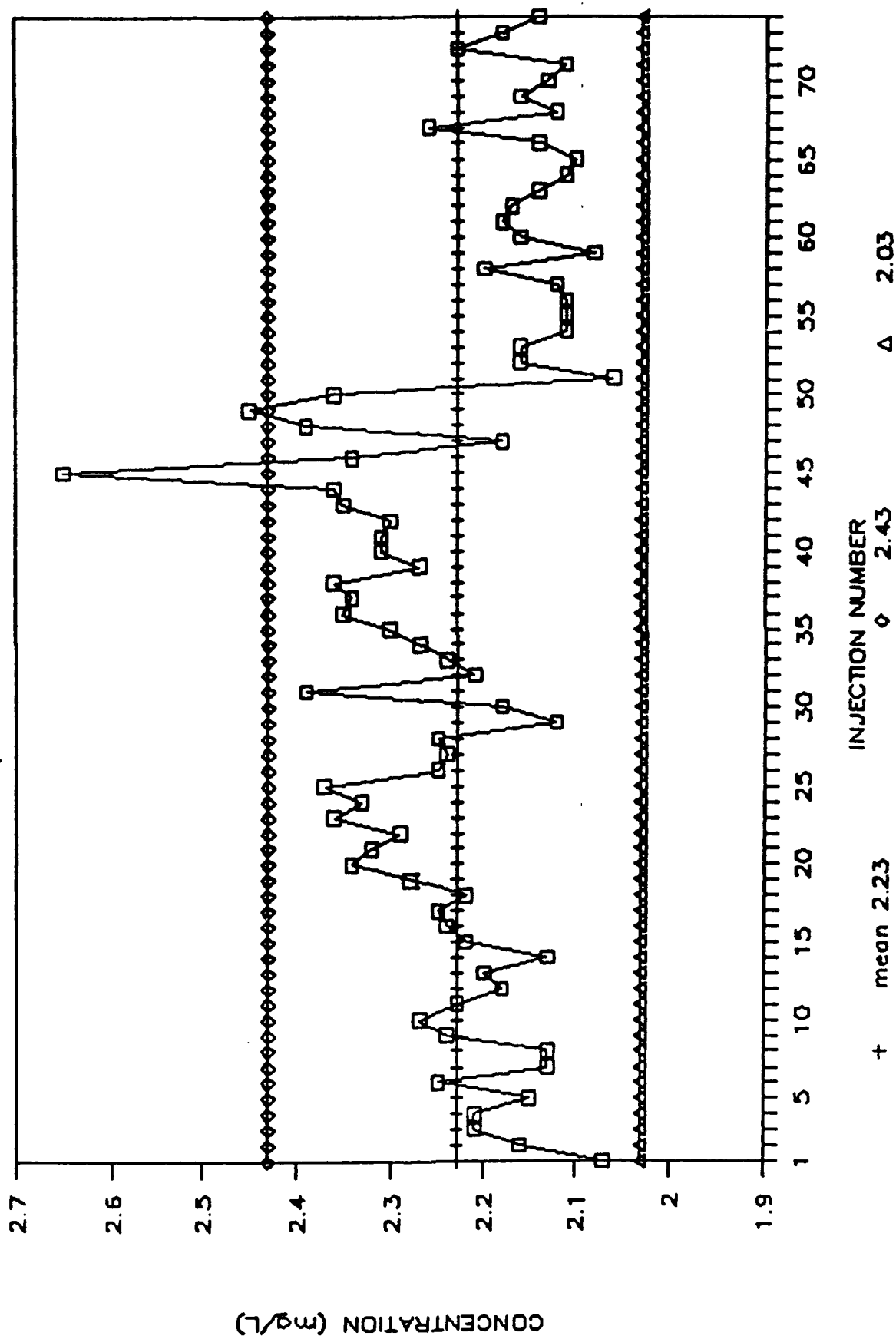


Fig. A8

4-AMINO 2,6 DNT VARIABILITY IN THE MTPS

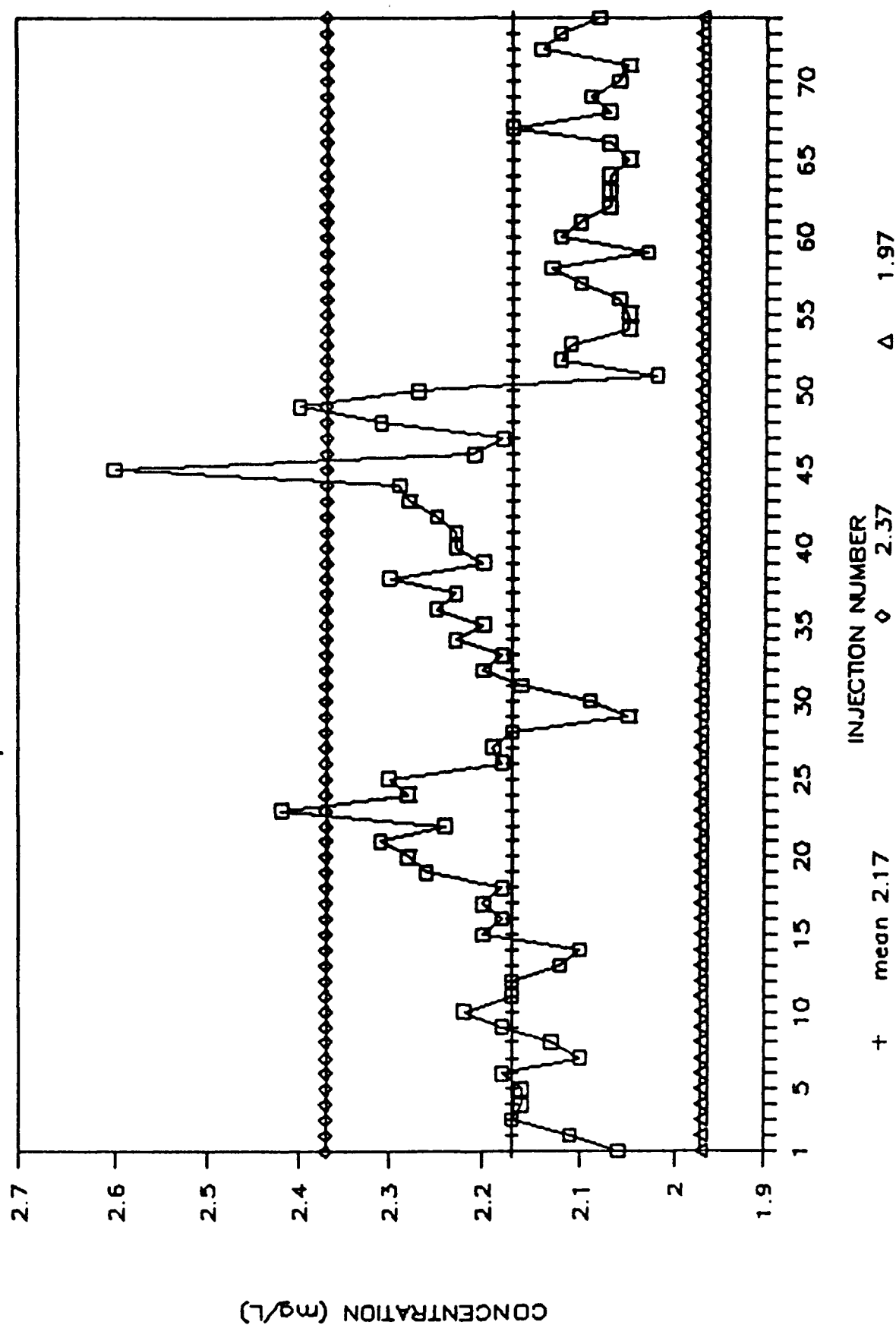


Fig. A9
DNB VARIABILITY IN THE MTPS

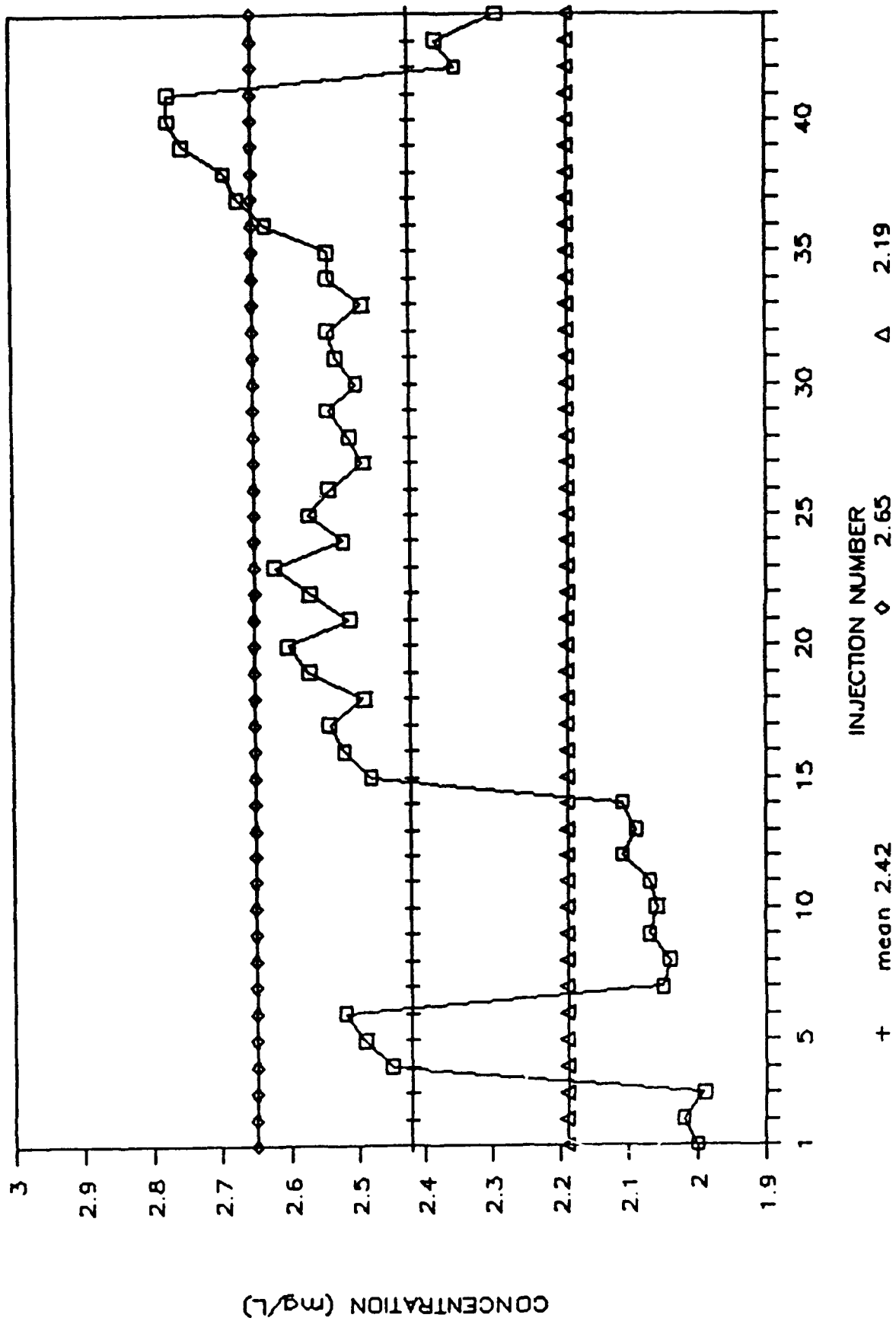
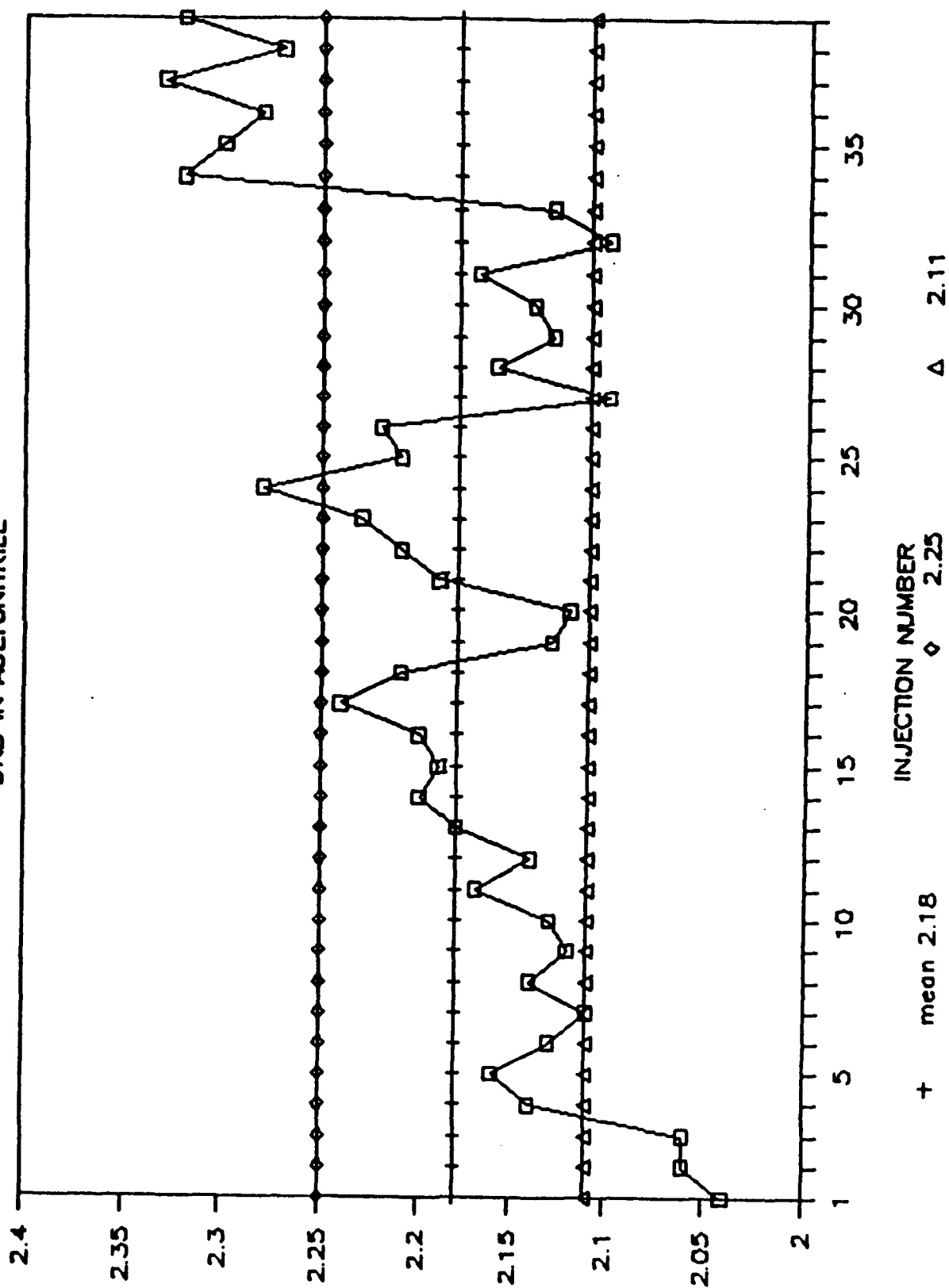


TABLE A1
PERCENT RECOVERY BY SITE

| COMPOUND | RADFORD | | MILAN | |
|--------------|-----------|-----|-----------|------|
| | %RECOVERY | STD | %RECOVERY | STD |
| HMX | 108.4 | 4.5 | 102.07 | 4.39 |
| TNB | 111.0 | 2.0 | 110.56 | 8.90 |
| RDX | 105.35 | 1.9 | 104.06 | 7.34 |
| DNB | 93.85 | 1.3 | NONE | |
| TNT | 99.50 | 1.2 | 108.91 | 6.74 |
| 2,4 DNT | 103.45 | 1.3 | 107.24 | 6.84 |
| 2,6 DNT | 100.95 | 1.9 | 107.02 | 8.81 |
| 2-AM 4,6 DNT | 104.10 | 1.2 | NONE | |
| 4-AM 2,6 DNT | 104.05 | 2.5 | NONE | |

| COMPOUND | PUEBLO | | ANNISTON | |
|--------------|-----------|-------|-----------|-------|
| | %RECOVERY | STD | %RECOVERY | STD |
| HMX | NONE | | 86.45 | 8.68 |
| RDX | NONE | | 84.06 | 8.15 |
| TNB | 91.20 | 7.28 | 95.69 | 11.45 |
| TNT | 94.04 | 8.63 | 98.99 | 12.43 |
| 2,4 DNT | 77.07 | 4.48 | 78.84 | 7.64 |
| 2,6 DNT | 77.89 | 4.97 | 79.78 | 8.59 |
| 2-AM 4,6 DNT | 57.63 | 14.43 | 73.48 | 21.87 |
| 4-AM 2,6 DNT | 86.93 | 14.80 | 144.31 | 42.35 |

Fig. A10
DNB IN ACETONITRILE



APPENDIX B

CRITERIA OF DETECTION

a. Explosives in Soil.

A criterion of detection (minimum accurate quantitation limit) was calculated from data of analysis of soil extracts in which the extraction and analysis steps were performed in triplicate and repeated in their entirety on four separate days. Criterion of detection of soil extracts was determined on a single soil type (Milan Soil). The soil was ground and sub-samples were spiked with 0.0, 0.4, 0.8, 1.63, 3.13, 6.25, 12.5, 25, and 50 mg/kg of a mixture of HMX, TNB, RDX, TNT, 2,4-DNT, 2,6-DNT, 2-AM, and 4-AM. For purposes of calculation the concentration of the explosives spiked onto the soil was assumed to be the "target concentration" in the soil at the time of analysis. The soils were extracted in the manner used for samples and the extracts analyzed. Target concentrations and the analytically derived values of the replicates were entered into the USATHAMA program for calculation of criteria of detection (Tables F1 - F8). This program generates a two dimensional plot with found values (analytically derived) as the dependent variable and target concentration as the independent variable (Figures F1 - F8). Linear regression of this relationship produces an equation in the form $Y = mx + b$ with;

Y = the found concentration

b = the found concentration intercept

m = the slope of the line

The variance about the regression line is plotted, thus generating parallel lines above and below the regression line. At the point where the line representing the mean minus the variance contacts the ordinate, values of Y can no longer be reliably distinguished from zero (Figures F9 - F16). Thus, criterion of detection is defined as the lowest concentration of analyte in an environmental sample which can be reliably distinguished from zero. Results of criterion of detection of soil extraction studies are summarized in Table F9. The criterion of detection levels from soil are:

| Compound | Criterion of Detection |
|----------|------------------------|
| HMX | 2.9 mg/kg |
| TNB | 2.4 mg/kg |
| RDX | 5.8 mg/kg |
| DNT | 6.1 mg/kg |
| 2,4-DNT | 5.7 mg/kg |
| 2,6-DNT | 5.2 mg/kg |
| 2-AM | 15.4 mg/kg |
| 4-AM | 14.6 mg/kg |

b. Explosives in Leachates.

In addition to the work done with soil extracts, criterion of detection was also performed for the leachates. The criterion of detection for these samples corresponds to the quantitation limit of the instrument because no sample preparation steps were employed.

The multipart standard containing HMX, TNB, RDX, TNT, 2,4-DNT, 2,6-DNT, 2-AM, and 4-AM was prepared at 1000 mg/L. This solution was diluted in a serial fashion to yield concentrations of 10, 5, 2.5, 1.25, 0.63, 0.32, 0.16, 0.08, 0.04, and 0.02 mg/L. These concentrations were analyzed in triplicate on four separate days and the results used to calculate the criterion of detection for each compound. Two separate criterion of detection studies were completed for the aqueous leachates and data from both studies are presented. Data from the first and second iteration of this work are identified by the small letter "a and b" after the table or figure number. For purposes of calculation the concentration of the explosives spiked into solution was the "target concentration". Target concentrations and the analytically derived values of the replicates were entered into the USATHAMA program for calculation of criteria of detection (Tables F10 - F17). This program generates a two dimensional plot with found values (analytically derived) as the dependent variable and target concentration as the independent variable (Figures F17 - F24). Linear regression of this relationship produces an equation in the form $Y = mx + b$ with;

Y = the found concentration
b = the found concentration intercept
m = the slope of the line

The variance about the regression line is plotted, thus generating parallel lines above and below the regression line. At the point where the line representing the mean minus the variance contacts the ordinate, values of Y can no longer be reliably distinguished from zero (Figures F25 - F32). Thus, criterion of detection is defined as the lowest concentration of analyte in an environmental sample which can be reliably distinguished from zero. Results of criterion of detection of leachate studies are summarized in Table F18. The criterion of detection levels for water and solvent are:

| Compound | Criterion of Detection |
|----------|------------------------|
| HMX | 0.14 mg/L |
| TNB | 0.14 mg/L |
| RDX | 0.12 mg/L |
| DNB | 0.15 mg/L |
| TNT | 0.09 mg/L |
| 2,4 DNT | 0.17 mg/L |
| 2,6 DNT | 0.36 mg/L |
| 2-AM | 0.14 mg/L |
| 4-AM | 0.14 mg/L |

Table Fl

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
Method Number: 1
Compound: HMX

Units of Measure: mg/Kg
Laboratory: RW
Analysis Date: 03/18/92
Matrix: SF

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
Y = (-0.24876344) + (0.854201200)X Y = (0.846765184)X

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 231.3894150 | 94 | 2.461589521 | 235.1184280 | 95 | 2.474930821 |
| Total Error: | 227.2558750 | 88 | 2.582453125 | 227.2558750 | 88 | 2.582453125 |
| Lack of Fit: | 4.133540000 | 6 | 0.688923333 | 7.862553000 | 7 | 1.123221857 |

LOF F-Ratio(F): 0.266770896 LOF F-Ratio(F): 0.434943754
Critical 95% F: 2.25 Critical 95% F: 2.17

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 1.514880108 Critical 95% F: 4

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| Target Value | Found Concentration |
|--------------|--|
| 1: 50 | 41.500000 43.200000 42.300000 45.600000 46.500000
48.500000 40.400000 41.900000 42.400000 39.700000
38.900000 39 |
| 2: 25 | 20.900000 21.400000 21.200000 22.900000 22.700000
23 21.700000 21.700000 21.800000 19.400000
19.400000 19.500000 |
| 3: 12.500000 | 10.700000 10.600000 10.300000 9.940000 9.260000
12.500000 10.400000 10.300000 9.600000 10
14.300000 1.200000 |
| 4: 6.2500000 | 5.2000000 4.5400000 4.8000000 5 5.0900000
5.1900000 5.1000000 4.8000000 5.1000000 5.1900000
4.9000000 4.9000000 |

Table F1 (Cont.)

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
 Method Number: 1
 Compound: HMX

Units of Measure: mg/Kg
 Laboratory: RW
 Analysis Date: 03/18/92
 Matrix: SF

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| | Target Value | Found Concentration | | | | |
|----|--------------|---------------------|-----------|-----------|-----------|-----------|
| 5: | 3.1300000 | 2.6700000 | 2.4800000 | 2.4800000 | 2.7000000 | 2 |
| | | 2.3000000 | 2.7700000 | 2.6700000 | 2.4800000 | 2.5000000 |
| | | 2.5000000 | 2.6000000 | | | |
| 6: | 1.5600000 | 1.1200000 | 1.9000000 | 1.2100000 | 1.0300000 | 1.2200000 |
| | | 1.8000000 | 1.3200000 | 0.9300000 | 0.6400000 | 1.4000000 |
| | | 1.1000000 | 0.9900000 | | | |
| 7: | 0.8000000 | 0.8400000 | 0.7000000 | 0.6500000 | 0.6400000 | 0.7300000 |
| | | 0.5400000 | 0.4400000 | 0.5400000 | 0.5400000 | 0.6400000 |
| | | 0.2500000 | 0 | | | |
| 8: | 0.4000000 | 0.4400000 | 0.6900000 | 0.6100000 | 0 | 0 |
| | | 0 | 0 | 0 | 0 | 0 |
| | | 0 | 0 | | | |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Table F2

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
 Method Number: 1
 Compound: TNB

Units of Measure: mg/Kg
 Laboratory: RW
 Analysis Date: 03/18/92
 Matrix: SF

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (0.141512116) + (0.905973870)X$ $Y = (0.910203938)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 176.8768300 | 94 | 1.881668404 | 178.0835540 | 95 | 1.874563726 |
| Total Error: | 168.7549830 | 88 | 1.917670261 | 168.7549830 | 88 | 1.917670261 |
| Lack of Fit: | 8.121847000 | 6 | 1.353641167 | 9.328571000 | 7 | 1.332653000 |

LOF F-Ratio(F): 0.705877957 LOF F-Ratio(F): 0.694933340
 Critical 95% F: 2.25 Critical 95% F: 2.17

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 0.641305342 Critical 95% F: 4

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| | Target Value | Found Concentration |
|----|--------------|--|
| 1: | 50 | 45.600000 47.500000 46.100000 43.300000 43.800000
51.600000 42 45.300000 46.100000 45.900000
44.900000 45.400000 |
| 2: | 25 | 23 22.900000 22.900000 23.400000 23.500000
23.500000 18.900000 21.300000 20.400000 23.900000
23.700000 23.800000 |
| 3: | 12.500000 | 11.900000 11.700000 11.300000 10.900000 7.4700000
5.6300000 12.900000 11.700000 11.200000 11.600000
12 12.700000 |
| 4: | 6.2500000 | 5.9100000 5.9100000 6.0900000 5.7000000 5.3000000
5.6800000 5.9100000 5.8600000 5.8000000 7
7.2000000 6.8000000 |

Table F2 (Cont.)

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
 Method Number: 1
 Compound: TNB

Units of Measure: mg/Kg
 Laboratory: RW
 Analysis Date: 03/18/92
 Matrix: SF

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| | Target Value | Found Concentration | | | | | |
|----|--------------|---------------------|-----------|-----------|-----------|-----------|--|
| 5: | 3.1300000 | 4.2000000 | 4.2000000 | 4.1000000 | 3.0400000 | 3.0400000 | |
| | | 2.7500000 | 3.1000000 | 2.2900000 | 2.2300000 | 2.8600000 | |
| | | 2.9800000 | 2.9200000 | | | | |
| 6: | 1.5600000 | 1.4800000 | 1.4800000 | 1.5400000 | 1.5400000 | 0.8500000 | |
| | | 1.0800000 | 1.2000000 | 1.5400000 | 2.8000000 | 1.3700000 | |
| | | 2.3000000 | 2.9000000 | | | | |
| 7: | 0.8000000 | 0.2300000 | 0.2200000 | 0.2100000 | 0.6200000 | 0.6200000 | |
| | | 0.5600000 | 0.7900000 | 0.5100000 | 0.3300000 | 0.9100000 | |
| | | 0.9100000 | 0.7900000 | | | | |
| 8: | 0.4000000 | 0.2900000 | 2.6000000 | 2.6000000 | 2 | 2 | |
| | | 0 | 0 | 0 | 0 | 0 | |
| | | 0 | 0 | | | | |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS

Table F3

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
Method Number: 1
Compound: RDX

Units of Measure: mg/Kg
Laboratory: RW
Analysis Date: 03/18/92
Matrix: SF

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
Y = (-0.11490761) + (0.744807248)X Y = (0.741372440)X

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 703.3546070 | 94 | 7.482495819 | 704.1502500 | 95 | 7.412107895 |
| Total Error: | 684.0883830 | 88 | 7.773731625 | 684.0883830 | 88 | 7.773731625 |
| Lack of Fit: | 19.26622400 | 6 | 3.211037333 | 20.06186700 | 7 | 2.865981000 |

LOF F-Ratio(F): 0.413062540 LOF F-Ratio(F): 0.368675063
Critical 95% F: 2.25 Critical 95% F: 2.17

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 0.106333905 Critical 95% F: 4

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| Target Value | Found Concentration |
|--------------|---|
| 1: 50 | 38.800000 39.900000 38.300000 25.900000 26.400000
42.100000 39.700000 40.200000 40.020000 39.500000
38.700000 38.700000 |
| 2: 25 | 19.500000 19.800000 20.400000 19.500000 19.500000
19.100000 6.210000 12 11.500000 21.400000
21.400000 21.100000 |
| 3: 12.500000 | 10 10.100000 9.250000 9.400000 9.100000
2.420000 11.700000 10.500000 10.100000 15.100000
10.800000 10.800000 |
| 4: 6.2500000 | 5.500000 6 4.800000 5 5.150000
4.850000 4.600000 4.600000 4.240000 4.400000
5.150000 4.850000 |

Table F3 (Cont.)

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
Method Number: 1
Compound: RDX

Units of Measure: mg/Kg
Laboratory: RW
Analysis Date: 03/18/92
Matrix: SF

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| | Target Value | Found Concentration | | | | | |
|----|--------------|---------------------|-----------|-----------|-----------|-----------|--|
| 5: | 3.1300000 | 2.2700000 | 2.1200000 | 2.1200000 | 2.4000000 | 0.6100000 | |
| | | 0.7600000 | 2.1200000 | 2.2700000 | 2.4300000 | 2.2000000 | |
| | | 2.3000000 | 2.8000000 | | | | |
| 6: | 1.5600000 | 2 | 1.7000000 | 1.2000000 | 0.4500000 | 1.0600000 | |
| | | 1.0600000 | 0.4500000 | 0 | 0 | 0.6100000 | |
| | | 1.6700000 | 1.0600000 | | | | |
| 7: | 0.8000000 | 0 | 0 | 0 | 0 | 0 | |
| | | 0 | 0 | 0 | 1.3000000 | 1 | |
| | | 1.7000000 | 0.9200000 | | | | |
| 8: | 0.4000000 | 0.9000000 | 0 | 0 | 0 | 0 | |
| | | 0 | 0 | 0 | 0 | 0 | |
| | | 0 | 0 | | | | |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Table F4

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
 Method Number: 1
 Compound: TNT

Units of Measure: mg/Kg
 Laboratory: RW
 Analysis Date: 03/19/92
 Matrix: SF

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.03971536) + (0.884832944)X$ $Y = (0.883644807)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 1095.426110 | 94 | 11.65346926 | 1095.521060 | 95 | 11.53180063 |
| Total Error: | 1069.960770 | 88 | 12.15864511 | 1069.960770 | 88 | 12.15864511 |
| Lack of Fit: | 25.46534000 | 6 | 4.244223333 | 25.56029000 | 7 | 3.651470000 |

LOF F-Ratio(F): 0.349070418 LOF F-Ratio(F): 0.300318824
 Critical 95% F: 2.25 Critical 95% F: 2.17

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 0.008147788 Critical 95% F: 4

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| | Target Value | Found Concentration |
|----|--------------|---|
| 1: | 50 | 50.600000 46.800000 51.200000 28.300000 27.200000
56.700000 45.700000 47.700000 47.700000 40.200000
41.300000 41.400000 |
| 2: | 25 | 20.700000 19.700000 20.600000 22.400000 23.700000
23.100000 14.800000 25.500000 26.300000 24.600000
23.800000 25.300000 |
| 3: | 12.500000 | 12.600000 10.800000 10.500000 13.200000 6.4400000
10.400000 11.300000 12.300000 11.600000 14.100000
13.700000 17.800000 |
| 4: | 6.2500000 | 8.3000000 3.7000000 7.7000000 5.7000000 5.6300000
5.9200000 4.2000000 5 5.3400000 5.9200000
5.5600000 5.5600000 |

Table F4 (Cont.)

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
Method Number: 1
Compound: TNT

Units of Measure: mg/Kg
Laboratory: RW
Analysis Date: 03/19/92
Matrix: SF

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| | Target Value | Found Concentration | | | | | |
|----|--------------|---------------------|-----------|-----------|-----------|-----------|--|
| 5: | 3.1300000 | 2.1800000 | 2.7800000 | 2.7800000 | 2.2000000 | 1.5300000 | |
| | | 1.1600000 | 2.4800000 | 1.9700000 | 2.2600000 | 2 | |
| | | 2.1000000 | 1.8000000 | | | | |
| 6: | 1.6500000 | 1.2000000 | 1.4000000 | 1.5000000 | 1.8200000 | 1.5300000 | |
| | | 0.9400000 | 1.2400000 | 1.4600000 | 1.6000000 | 1.3100000 | |
| | | 0.9400000 | 0.9500000 | | | | |
| 7: | 0.8000000 | 0.2600000 | 0.6500000 | 0.5800000 | 0.5800000 | 0 | |
| | | 0 | 0 | 0 | 0 | 1 | |
| | | 0.7000000 | 0 | | | | |
| 8: | 0.4000000 | 0 | 0 | 0 | 0 | 0 | |
| | | 0 | 1.6500000 | 0 | 0 | 0 | |
| | | 0 | 0 | | | | |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Table F5

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
 Method Number: 1
 Compound: 2,4DNT

Units of Measure: mg/Kg
 Laboratory: RW
 Analysis Date: 03/19/92
 Matrix: SF

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.59402705) + (0.809804126)X$ $Y = (0.792047521)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 792.6388120 | 94 | 8.432327787 | 813.9022350 | 95 | 8.567391947 |
| Total Error: | 777.3167500 | 88 | 8.833144886 | 777.3167500 | 88 | 8.833144886 |
| Lack of Fit: | 15.32206200 | 6 | 2.553677000 | 36.58548500 | 7 | 5.226497857 |

LOF F-Ratio(F): 0.289101677 LOF F-Ratio(F): 0.591691626
 Critical 95% F: 2.25 Critical 95% F: 2.17

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 2.521655175 Critical 95% F: 4

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| | Target Value | Found Concentration |
|----|--------------|---|
| 1: | 50 | 41.400000 40.200000 41.300000 40.200000 42.900000
41.500000 26.700000 26.900000 43.200000 42.500000
45.700000 46.400000 |
| 2: | 25 | 20.200000 21.200000 20.400000 12.500000 10.500000
13.600000 23.700000 23.700000 23.900000 22.600000
20.600000 19.700000 |
| 3: | 12.500000 | 12.100000 10.300000 16 10.200000 9.6200000
9.4700000 9.4200000 6.4000000 6.7100000 11.400000
11.800000 10.900000 |
| 4: | 6.2500000 | 4.5600000 4.8700000 5.3300000 2.9000000 5
2.9500000 4.7100000 3.1800000 4.2500000 2.8000000
3.4000000 3.2000000 |

Table F5 (Cont.)

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
 Method Number: 1
 Compound: 2,4DNT

Units of Measure: mg/Kg
 Laboratory: RW
 Analysis Date: 03/19/92
 Matrix: SF

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| | Target Value | Found Concentration | | | | |
|----|--------------|---------------------|-----------|-----------|-----------|-----------|
| 5: | 3.1300000 | 2 | 2.1000000 | 1.8000000 | 2.7200000 | 1.7200000 |
| | | 2.4900000 | 1.9000000 | 1.0300000 | 1.0300000 | 1.2600000 |
| | | 1.6400000 | 0.0300000 | | | |
| 6: | 1.5600000 | 0.5700000 | 0.5700000 | 0.5700000 | 0 | 0 |
| | | 0 | 0.2600000 | 0.4900000 | 0 | 1.4000000 |
| | | 1.4000000 | 1.5000000 | | | |
| 7: | 0.8000000 | 0 | 0 | 0.8000000 | 0 | 0 |
| | | 0 | 0 | 0 | 0 | 0 |
| | | 0 | 0 | | | |
| 8: | 0.4000000 | 0 | 0 | 0 | 0 | 0 |
| | | 0 | 0.9900000 | 0 | 0 | 0 |
| | | 0 | 0 | | | |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Table F6

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
 Method Number: 1
 Compound: 2,6DNT

Units of Measure: mg/Kg
 Laboratory: RW
 Analysis Date: 03/19/92
 Matrix: SF

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.58428181) + (0.824346024)X$ $Y = (0.806880723)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 681.4978330 | 94 | 7.249976947 | 702.0693100 | 95 | 7.390203263 |
| Total Error: | 643.8581280 | 88 | 7.316569636 | 643.8581280 | 88 | 7.316569636 |
| Lack of Fit: | 37.63970500 | 6 | 6.273284167 | 58.21118200 | 7 | 8.315883143 |

LOF F-Ratio(F): 0.857407840 LOF F-Ratio(F): 1.136582245
 Critical 95% F: 2.25 Critical 95% F: 2.17

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 2.837454126 Critical 95% F: 4

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| | Target Value | Found Concentration |
|----|--------------|---|
| 1: | 50 | 39.900000 31.400000 39.300000 44.300000 45.600000
47.200000 42.843000 44.500000 39.500000 42.200000
40.600000 24.600000 |
| 2: | 25 | 21.400000 21 20.400000 25.200000 24.100000
24.200000 23.300000 22.400000 20.200000 20
19.900000 14.500000 |
| 3: | 12.500000 | 10.900000 9.6200000 10.500000 9.8600000 6.6600000
5.2400000 4.2900000 10.300000 11.700000 9.8600000
11.100000 11.500000 |
| 4: | 6.2500000 | 2.8000000 3.4000000 3.2000000 4.4100000 5.1200000
5.2400000 3.3000000 3.8000000 2.2700000 4.4100000
4.7200000 4.5300000 |

Table F6 (Cont.)

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
Method Number: 1
Compound: 2,6DNT

Units of Measure: mg/Kg
Laboratory: RW
Analysis Date: 03/19/92
Matrix: SF

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| | Target Value | Found Concentration | | | | | |
|----|--------------|---------------------|-----------|-----------|-----------|-----------|--|
| 5: | 3.1300000 | 3.1000000 | 1.2000000 | 2.6300000 | 1.3000000 | 0.3700000 | |
| | | 0.4900000 | 1.0800000 | 1.3200000 | 1.4400000 | 3 | |
| | | 2.9000000 | 0 | | | | |
| 6: | 1.5600000 | 1.9000000 | 2.2000000 | 1.7000000 | 0 | 0 | |
| | | 0 | 0 | 0.2500000 | 0.6100000 | 0 | |
| | | 0 | 0 | | | | |
| 7: | 0.8000000 | 1.2000000 | 0 | 2 | 1.8000000 | 0 | |
| | | 0 | 0 | 0 | 0 | 0 | |
| | | 0 | 0 | | | | |
| 8: | 0.4000000 | 0 | 0 | 0 | 1.8000000 | 0 | |
| | | 0 | 0 | 0 | 0 | 0 | |
| | | 0 | 0 | | | | |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Table F7

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
 Method Number: 1
 Compound: 2-AM

Units of Measure: mg/Kg
 Laboratory: RW
 Analysis Date: 03/19/92
 Matrix: SF

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.73266610) + (0.786218675)X$ $Y = (0.764317883)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 5512.399130 | 94 | 58.64254394 | 5544.746050 | 95 | 58.36574789 |
| Total Error: | 5418.396520 | 88 | 61.57268773 | 5418.396520 | 88 | 61.57268773 |
| Lack of Fit: | 94.00261000 | 6 | 15.66710167 | 126.3495300 | 7 | 18.04993286 |

LOF F-Ratio(F): 0.254448884 LOF F-Ratio(F): 0.293148367
 Critical 95% F: 2.25 Critical 95% F: 2.17

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 0.551594761 Critical 95% F: 4

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| Target Value | Found Concentration |
|--------------|---|
| 1: 50 | 60.400000 66.600000 70.400000 21 21.100000
21.700000 35 47.500000 49.300000 17.400000
18 24.100000 |
| 2: 25 | 21.500000 15.300000 31.300000 38 33.400000
13.500000 12.800000 12.500000 15.300000 16.400000
18.300000 26.600000 |
| 3: 12.500000 | 10.900000 10.500000 9.8600000 6.6600000 5.2400000
4.2900000 10.300000 11.700000 9.8600000 7.6000000
6.1000000 7.1000000 |
| 4: 6.2500000 | 2.8000000 3.5000000 3.2000000 6.3500000 4.9400000
1.2400000 4.7000000 4.3000000 2.3000000 3.8800000
3.1800000 4.4100000 |

Table F7 (Cont.)

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
 Method Number: 1
 Compound: 2-AM

Units of Measure: mg/Kg
 Laboratory: RW
 Analysis Date: 03/19/92
 Matrix: SF

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| | Target Value | Found Concentration | | | | | |
|----|--------------|---------------------|-----------|-----------|-----------|-----------|--|
| 5: | 3.1300000 | 1.0600000 | 2.1200000 | 0.1800000 | 0.9000000 | 0.5400000 | |
| | | 0.3600000 | 1.0700000 | 1.7700000 | 1.6000000 | 1.6000000 | |
| | | 1.3000000 | 0 | | | | |
| 6: | 1.5600000 | 0.4000000 | 0.9000000 | 0.1000000 | 0.7000000 | 0 | |
| | | 0 | 0 | 0 | 0 | 0 | |
| | | 0 | 0 | | | | |
| 7: | 0.8000000 | 0 | 0 | 0 | 0 | 0 | |
| | | 0 | 0 | 0.3000000 | 0.1000000 | 0.6000000 | |
| | | 0 | 0 | | | | |
| 8: | 0.4000000 | 0 | 0 | 1.8200000 | 0 | 0 | |
| | | 0 | 0 | 0 | 0 | 0 | |
| | | 0 | 0 | | | | |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Table F8

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
 Method Number: 1
 Compound: 4-AM

Units of Measure: mg/Kg
 Laboratory: RW
 Analysis Date: 03/19/92
 Matrix: SF

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.63682244) + (0.745388360)X$ $Y = (0.726352519)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 4427.118830 | 94 | 47.09700883 | 4451.556370 | 95 | 46.85848811 |
| Total Error: | 4191.612510 | 88 | 47.63196034 | 4191.612510 | 88 | 47.63196034 |
| Lack of Fit: | 235.5063200 | 6 | 39.25105333 | 259.9438600 | 7 | 37.13483714 |

LOF F-Ratio(F): 0.824048665 LOF F-Ratio(F): 0.779620173
 Critical 95% F: 2.25 Critical 95% F: 2.17

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 0.518876689 Critical 95% F: 4

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| Target Value | Found Concentration |
|--------------|---|
| 1: 50 | 23 22.400000 25.600000 21.300000 37.600000
35.600000 32.261000 6.4400000 67.600000 47.700000
51.600000 47.700000 |
| 2: 25 | 12.500000 13.100000 12.800000 12.800000 21.300000
14.500000 19.800000 37.600000 35.600000 32.500000
28.700000 16.100000 |
| 3: 12.500000 | 9.9000000 8.3000000 7.7700000 9.1900000 6.5200000
7.7700000 12.700000 15.800000 14.700000 7.1000000
8.2000000 8.1000000 |
| 4: 6.2500000 | 3.4000000 2.5000000 3.4000000 2.9700000 2.6200000
2.7900000 3.1500000 2.9000000 1.9000000 3.1500000
2.9700000 4.2100000 |

Table F8 (Cont.)

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: SOIL EXTRACTION
 Method Number: 1
 Compound: 4-AM

Units of Measure: mg/Kg
 Laboratory: RW
 Analysis Date: 03/19/92
 Matrix: SF

TABLE OF DATA POINTS

Targets: 8

Measures per Target: 12

| | Target Value | Found Concentration | | | | |
|----|--------------|---------------------|-----------|-----------|---|-----------|
| 5: | 3.1300000 | 0.1300000 | 0 | 0.1300000 | 0 | 0 |
| | | 0 | 0 | 0 | 0 | 0.7000000 |
| | | 0.7000000 | 0.3400000 | | | |
| 6: | 1.5600000 | 0 | 0 | 0 | 0 | 0 |
| | | 0 | 0 | 0 | 0 | 0 |
| | | 0 | 0 | | | |
| 7: | 0.8000000 | 0 | 0 | 0 | 0 | 0 |
| | | 0 | 0 | 0 | 0 | 0 |
| | | 0 | 0 | | | |
| 8: | 0.4000000 | 0 | 0 | 0 | 0 | 0 |
| | | 0 | 0 | 0 | 0 | 0 |
| | | 0 | 0 | | | |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Figure F1
HMX

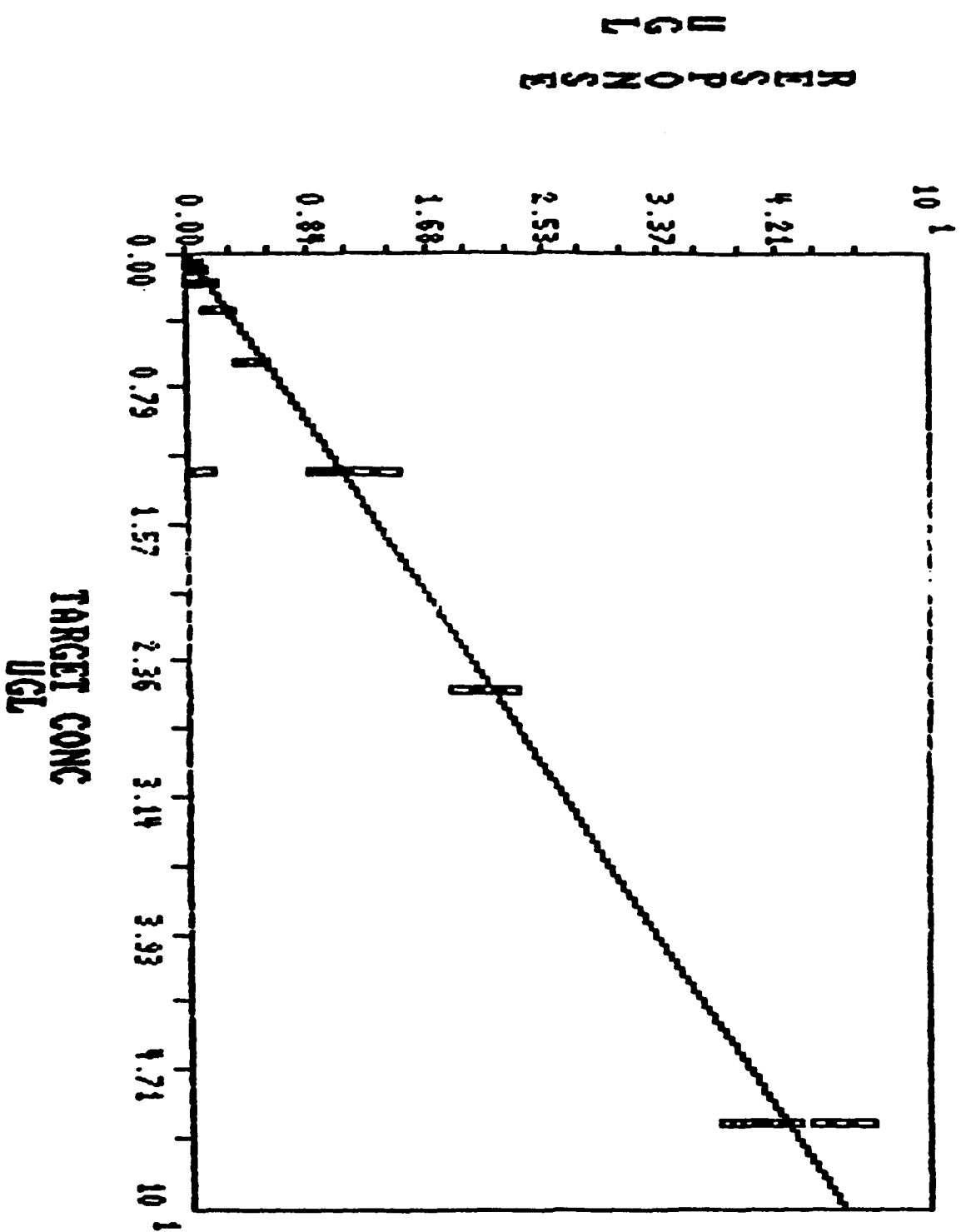


Figure F2
TNB

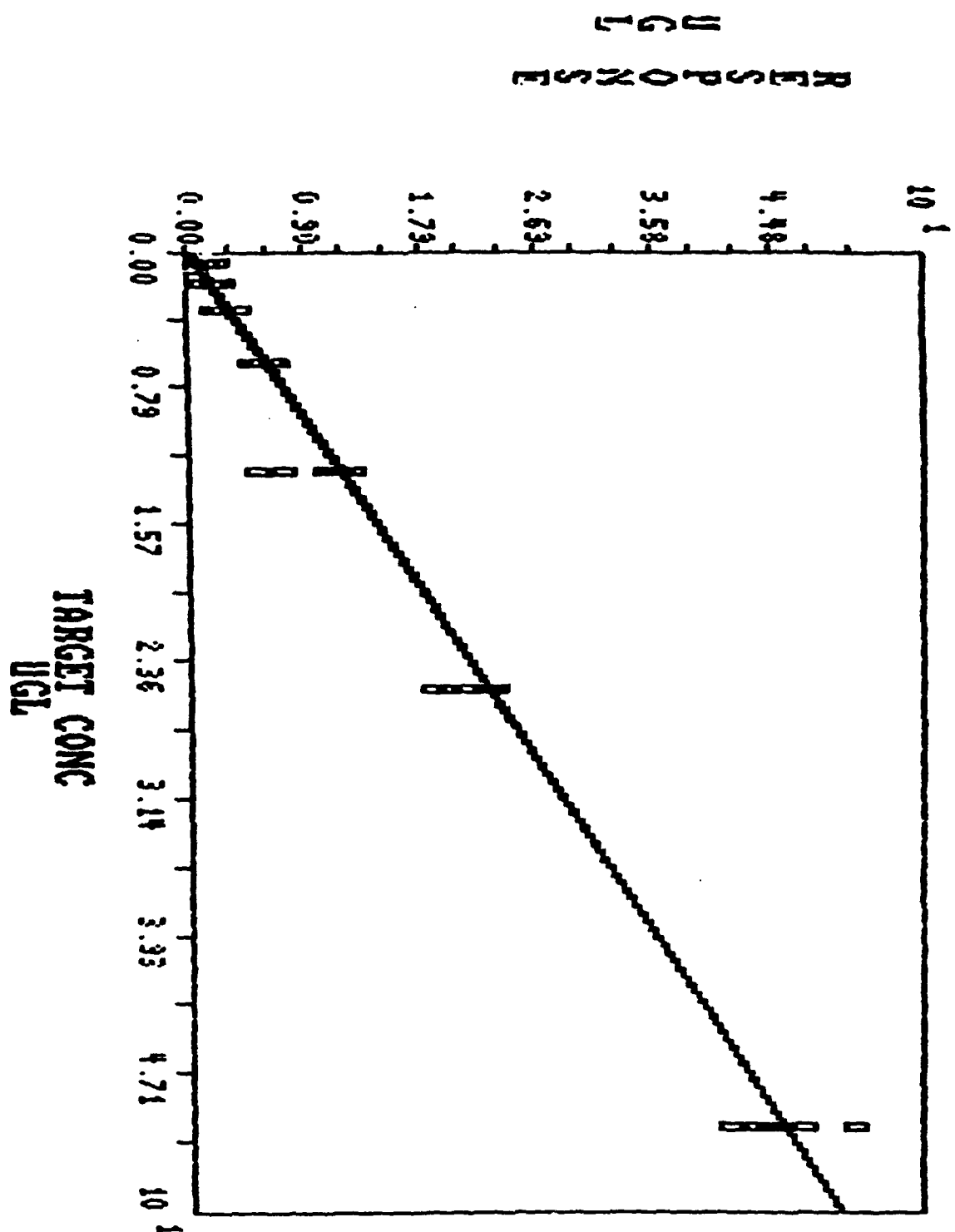


Figure F3
RDX

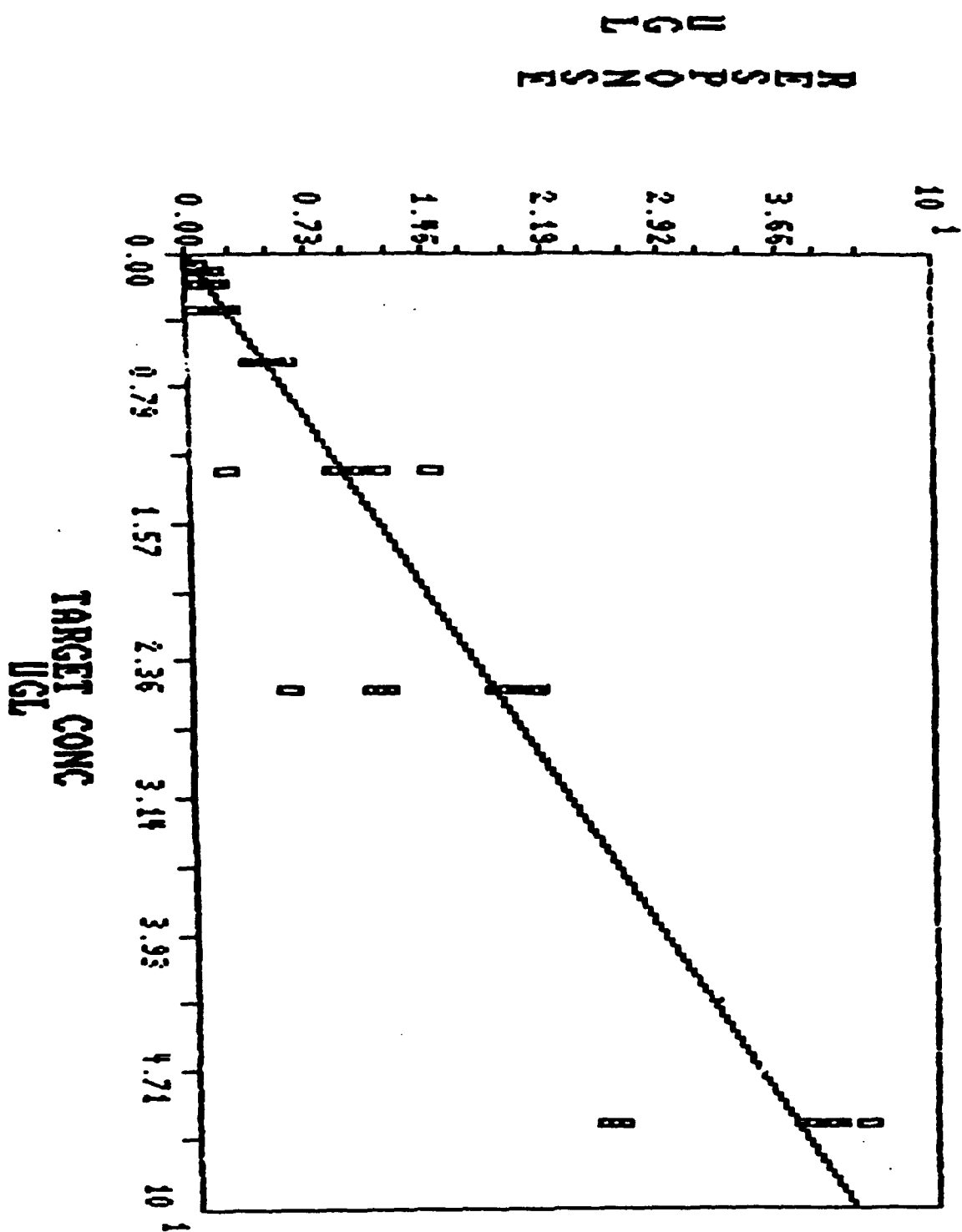


Figure F4
TNT

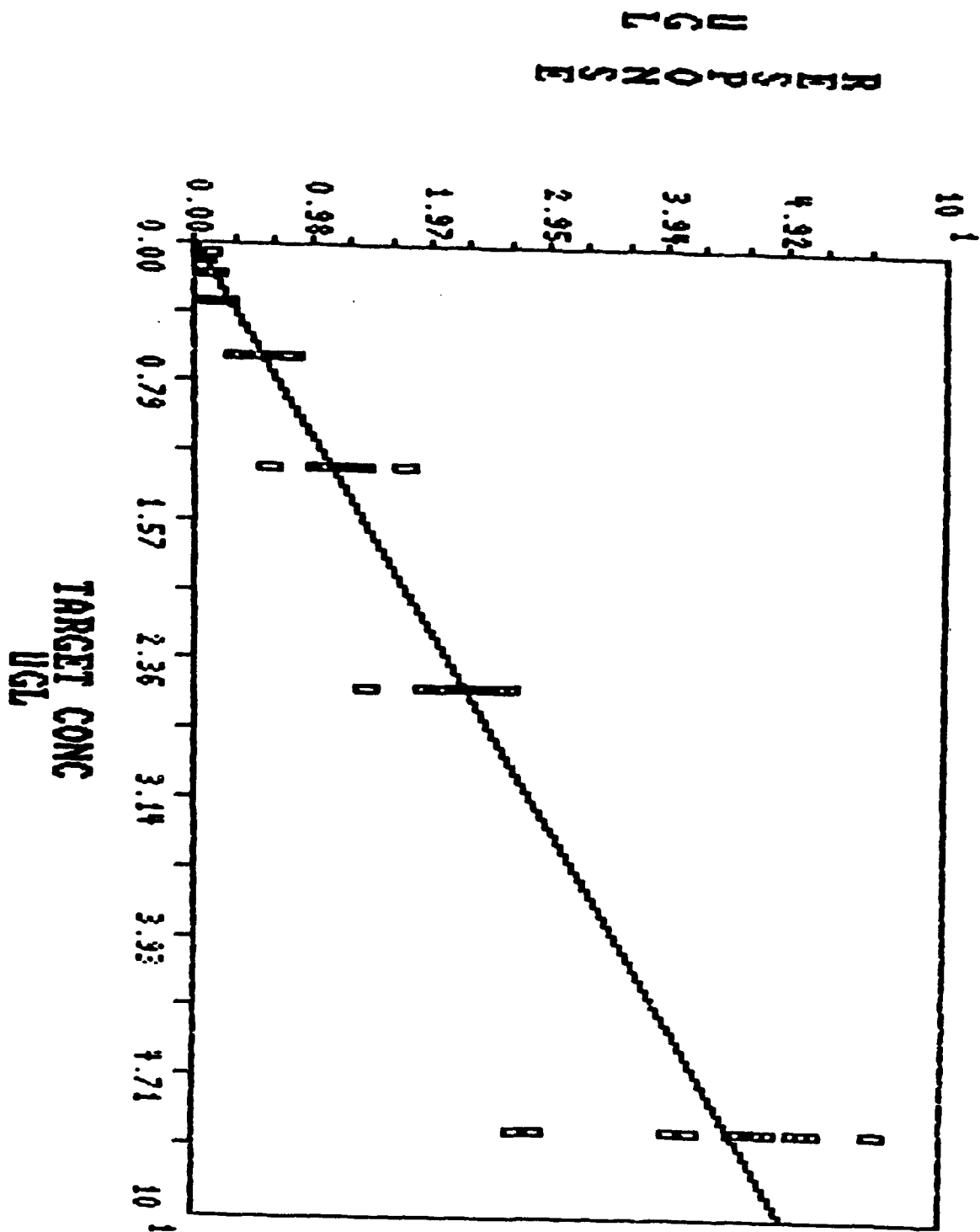


Figure F5
2,4DNT

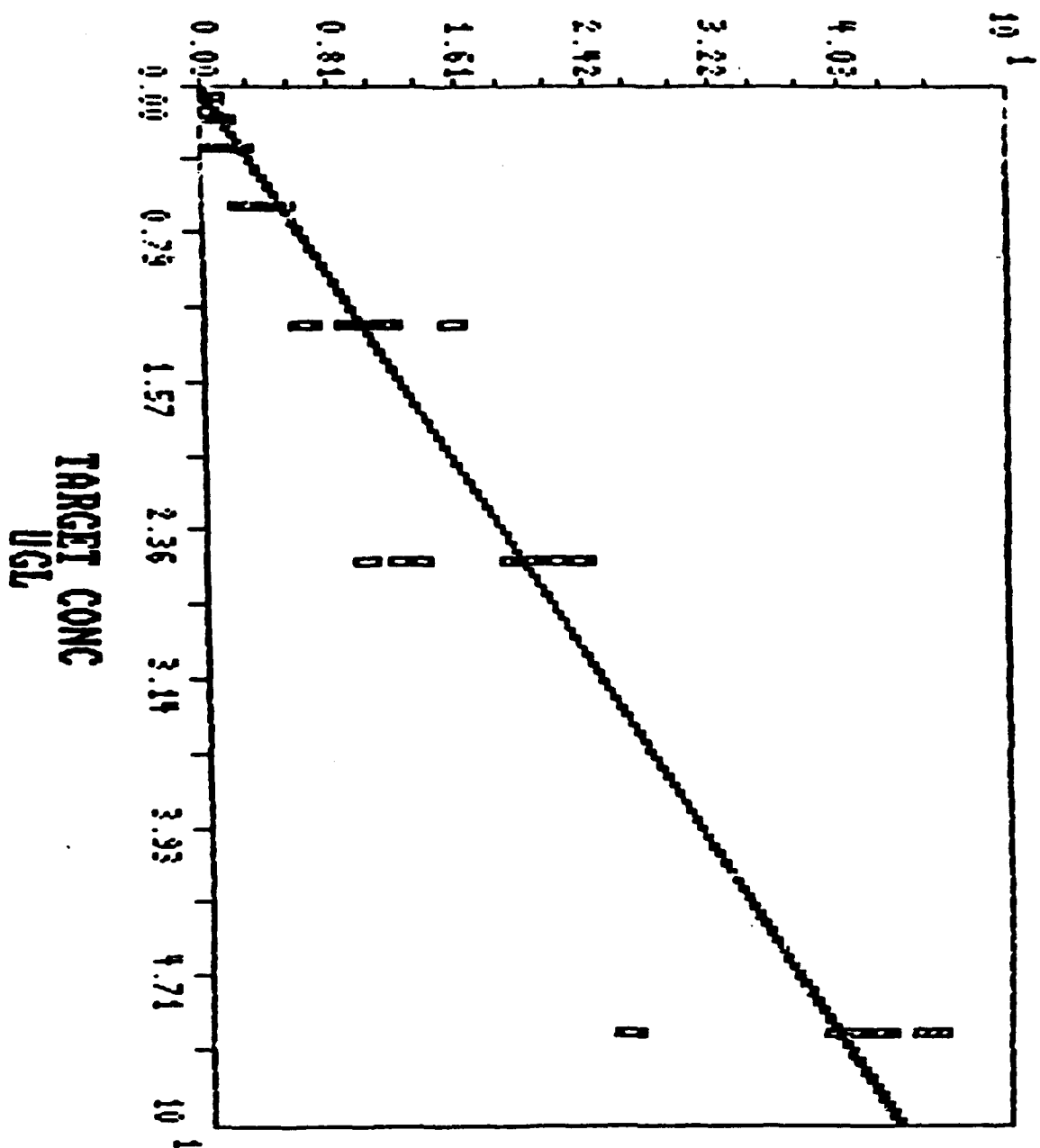


Figure F6
2,6DNT

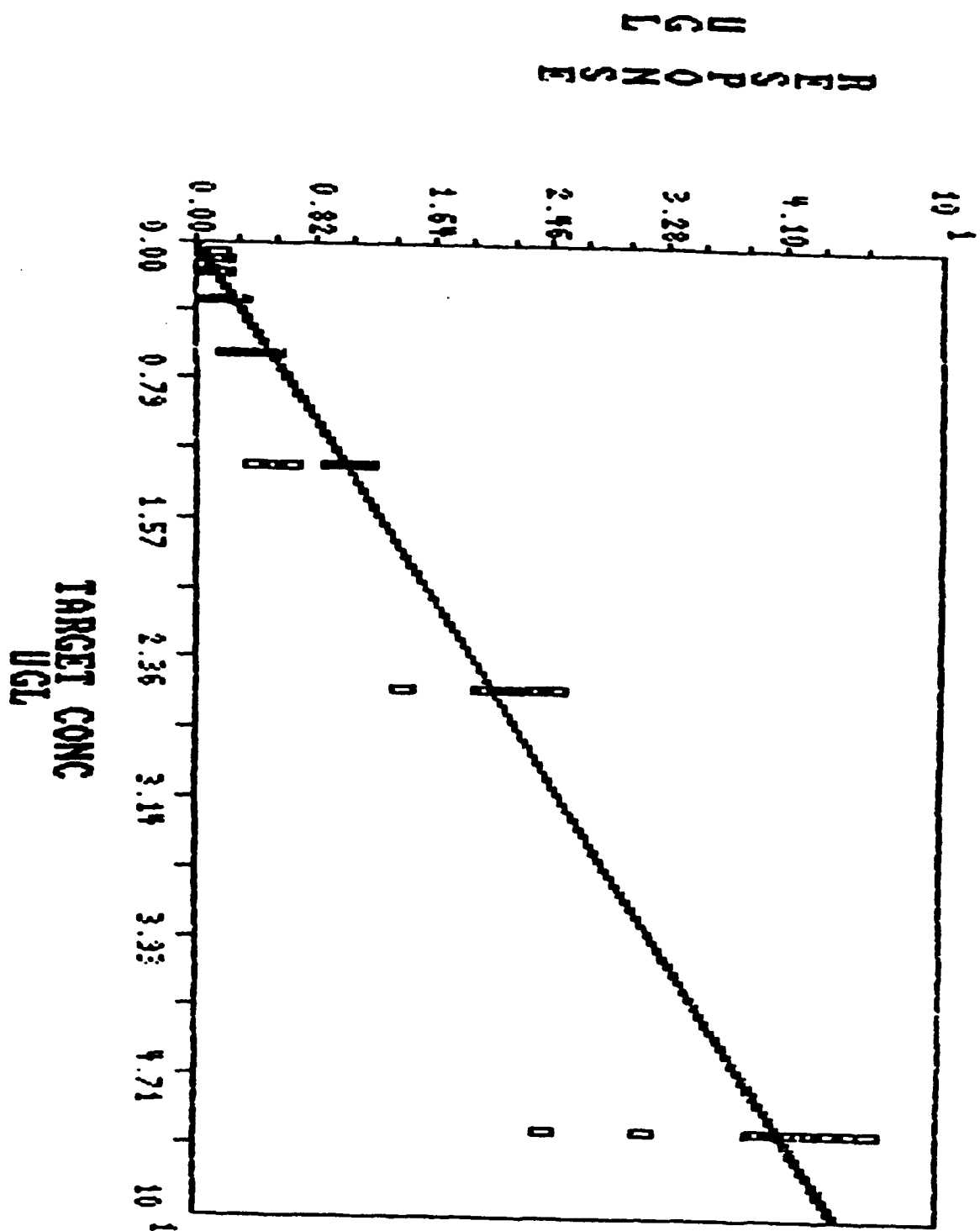


Figure F7
2-AM

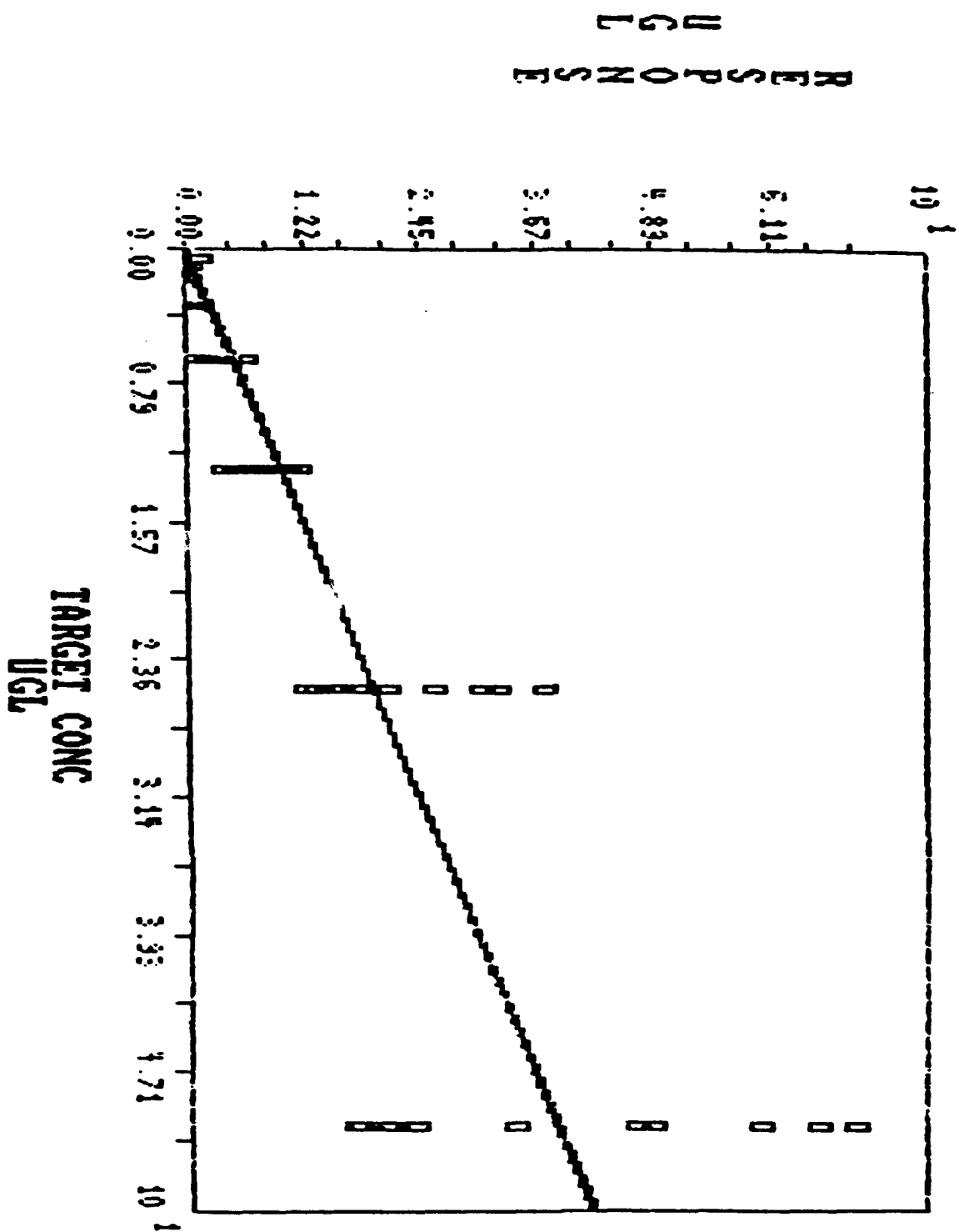


Figure F8

4-AM

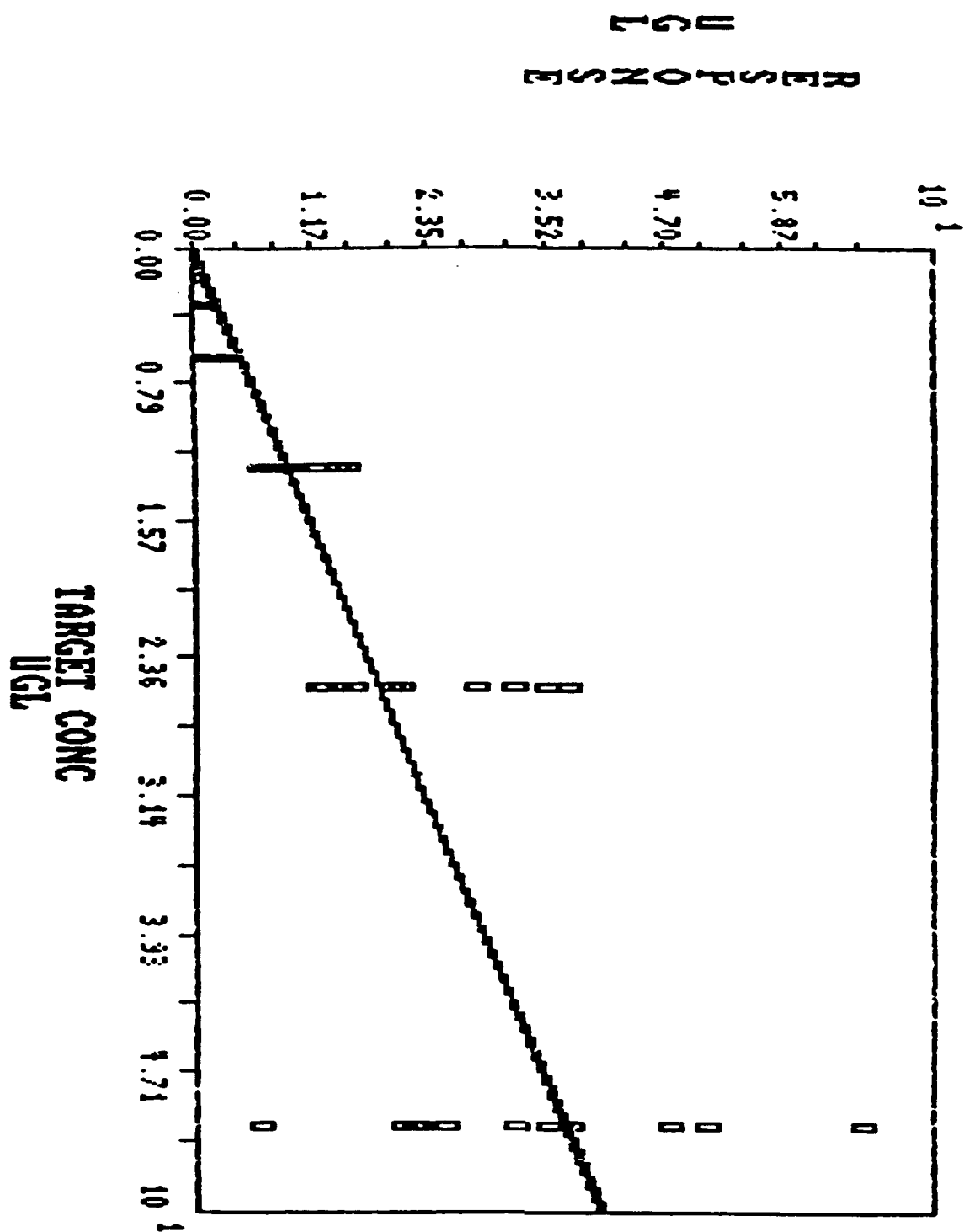


Figure F9

HMX

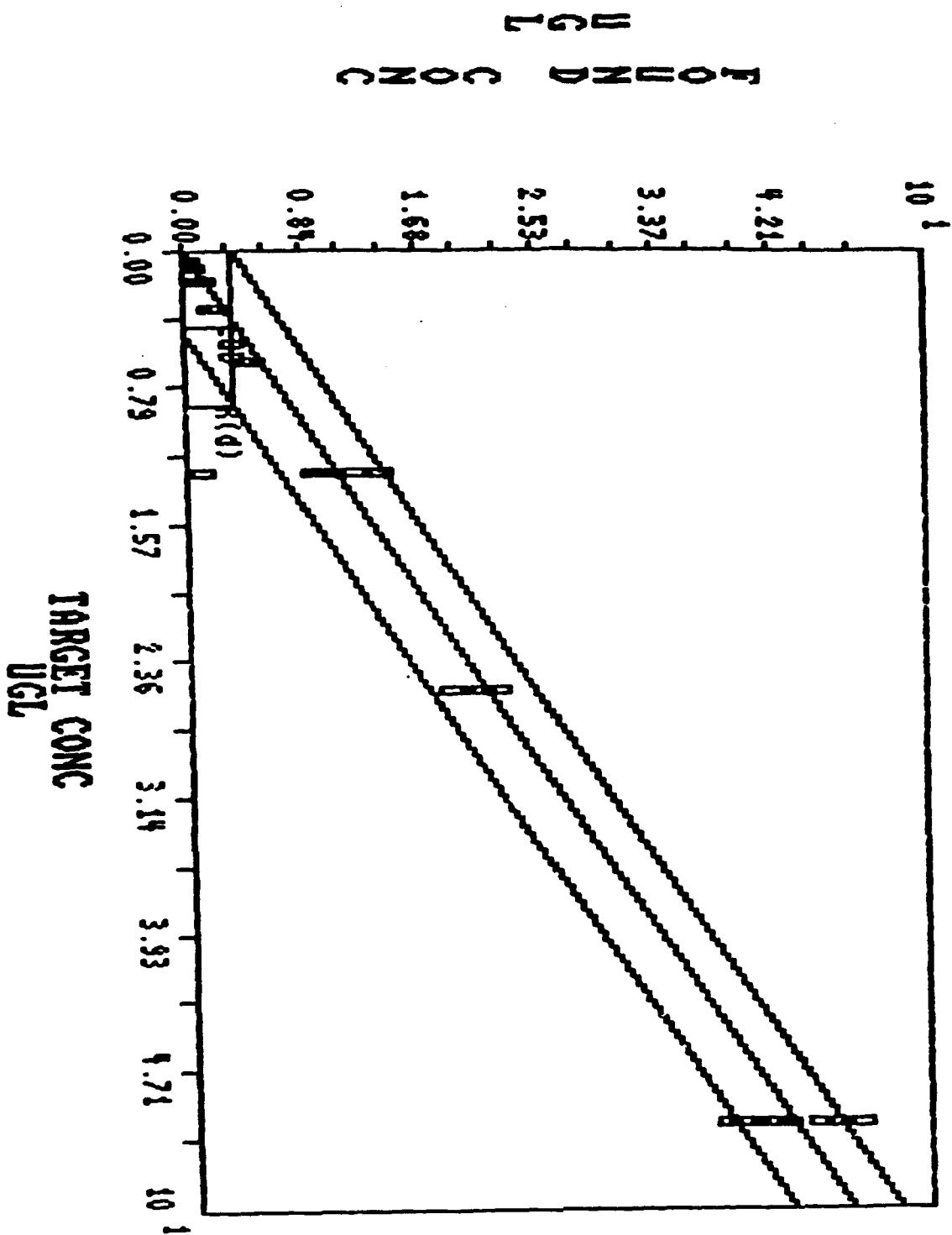
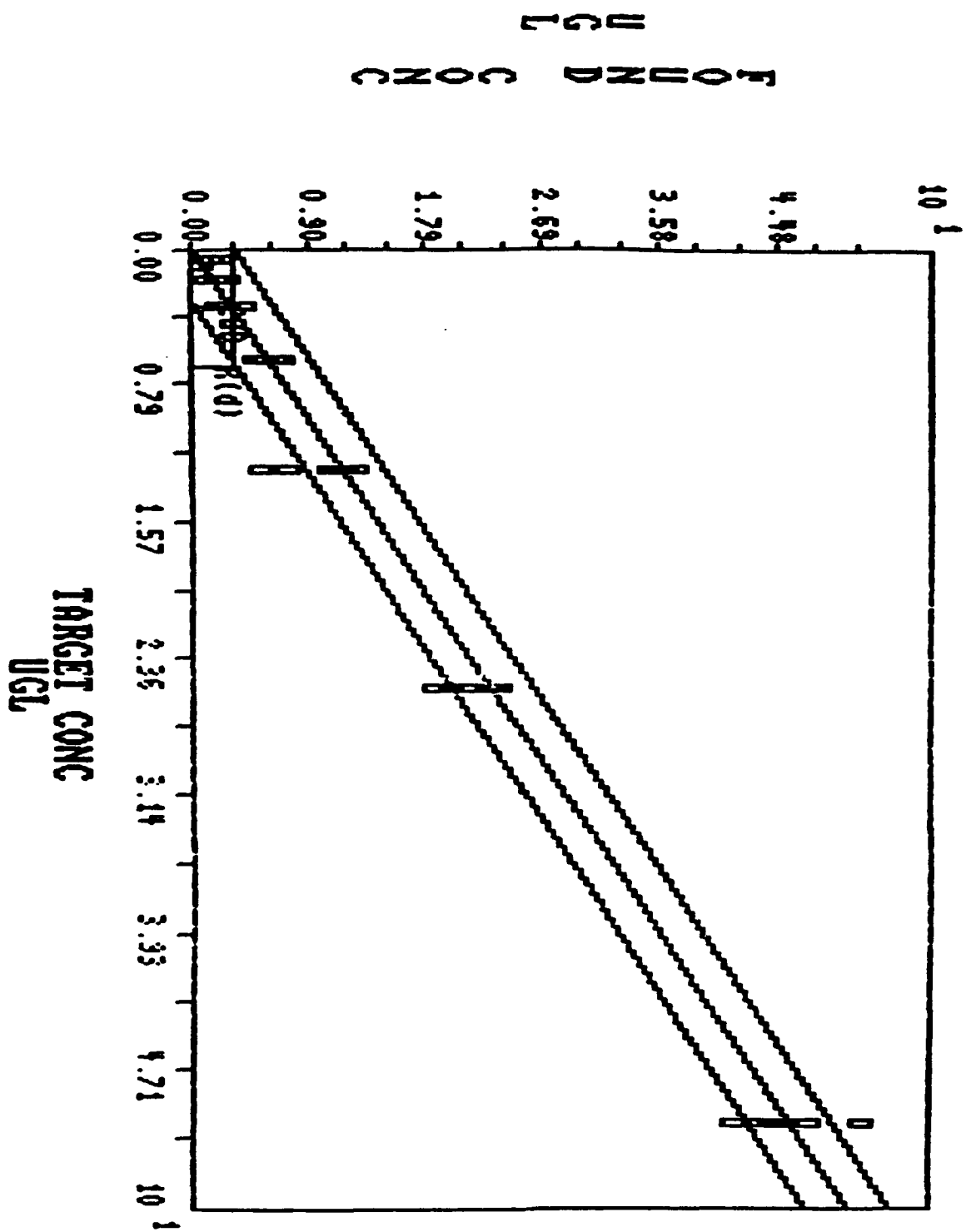


Figure F10
TNB



FDX



Figure F12

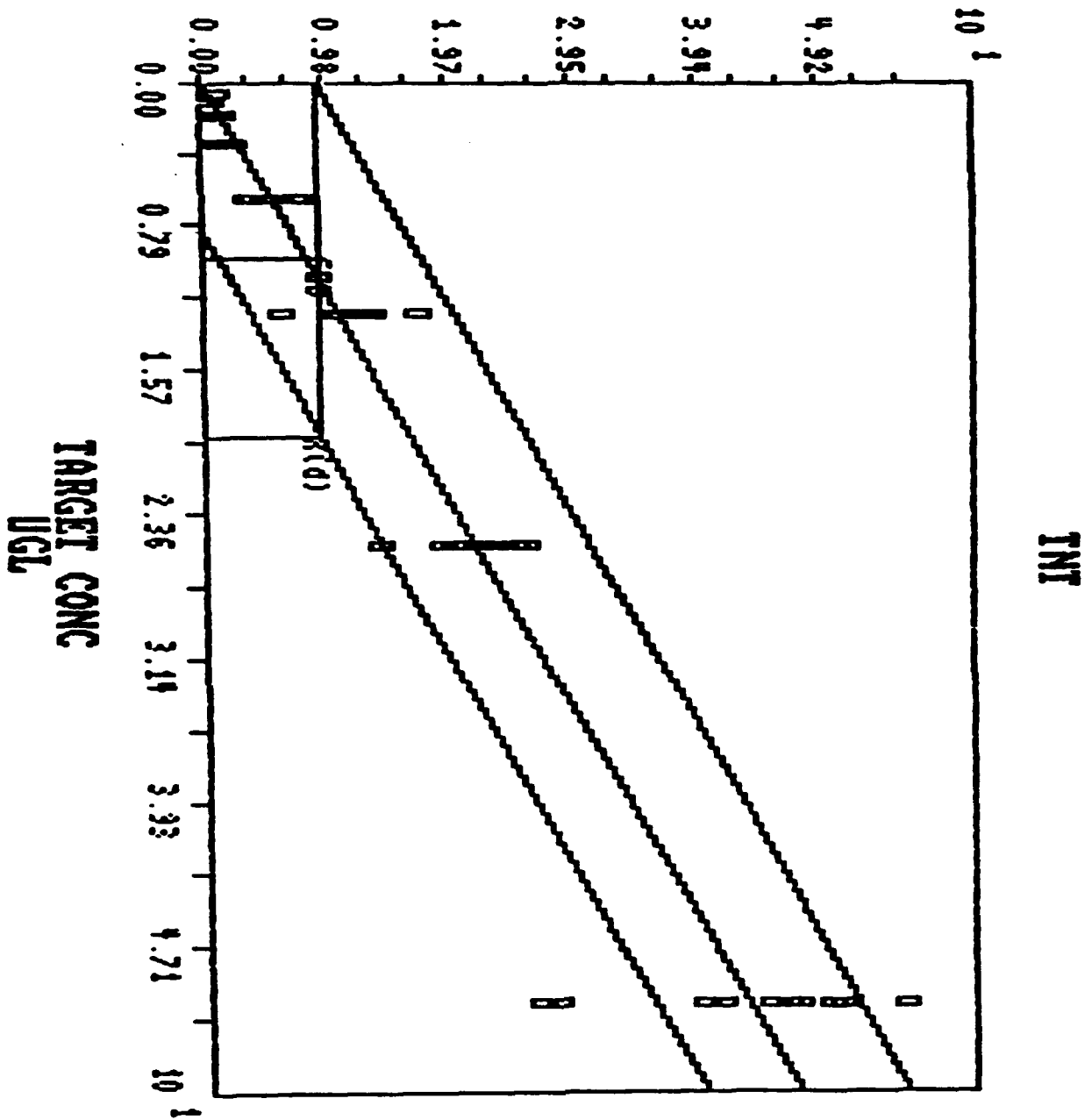


Figure F 13
2,4DNT

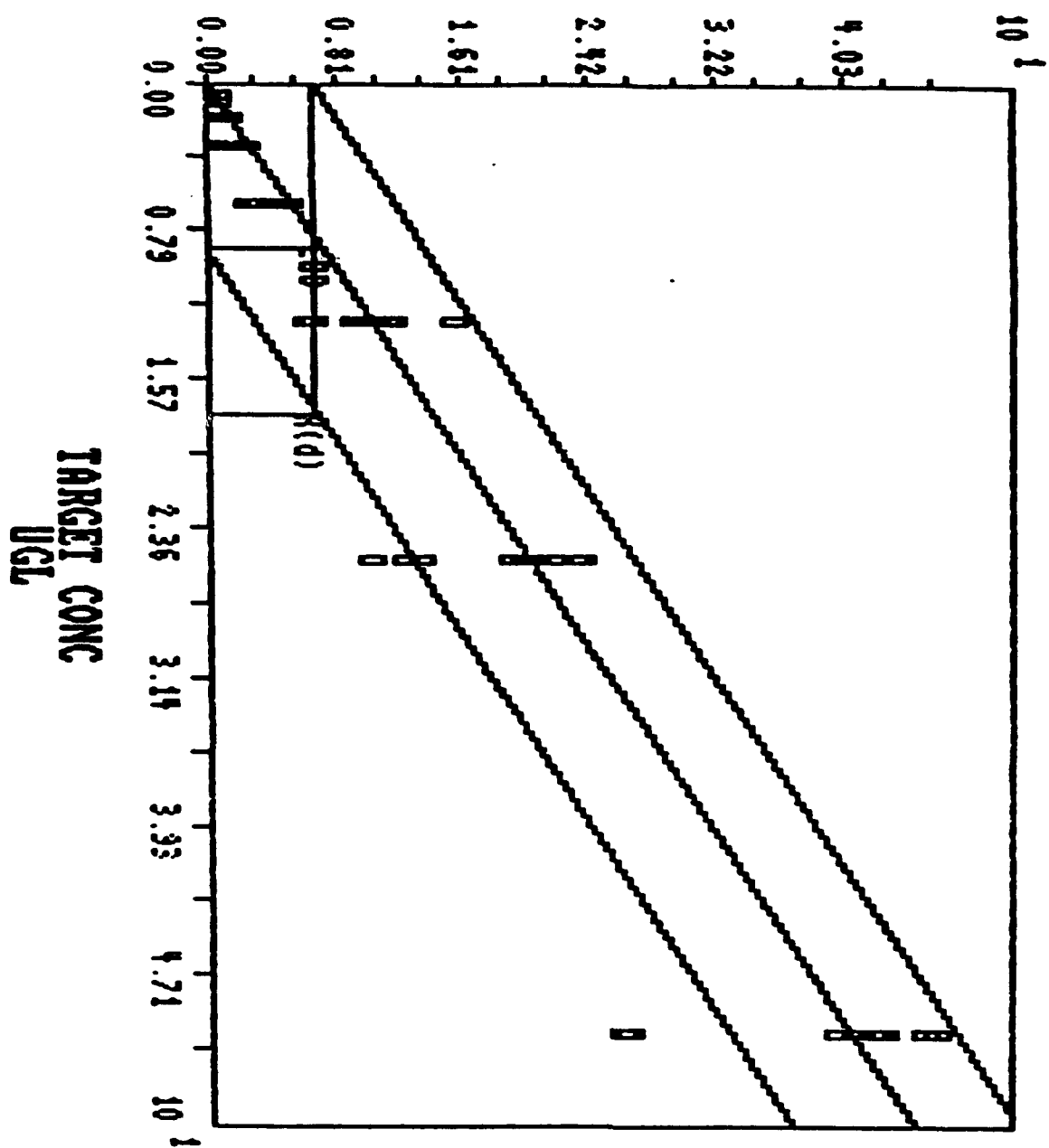


Figure F14
2,6DNT

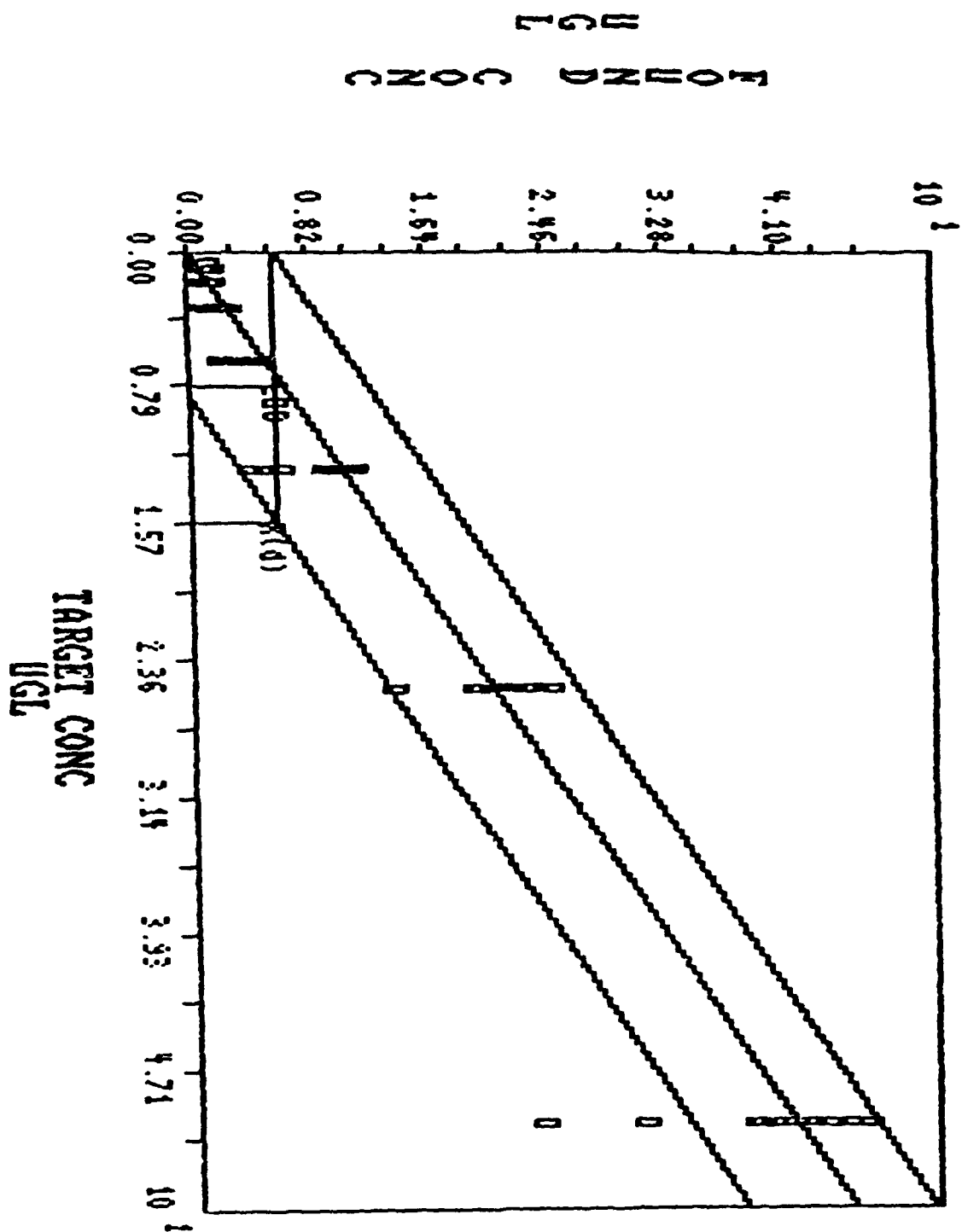


Figure F15

2-AM

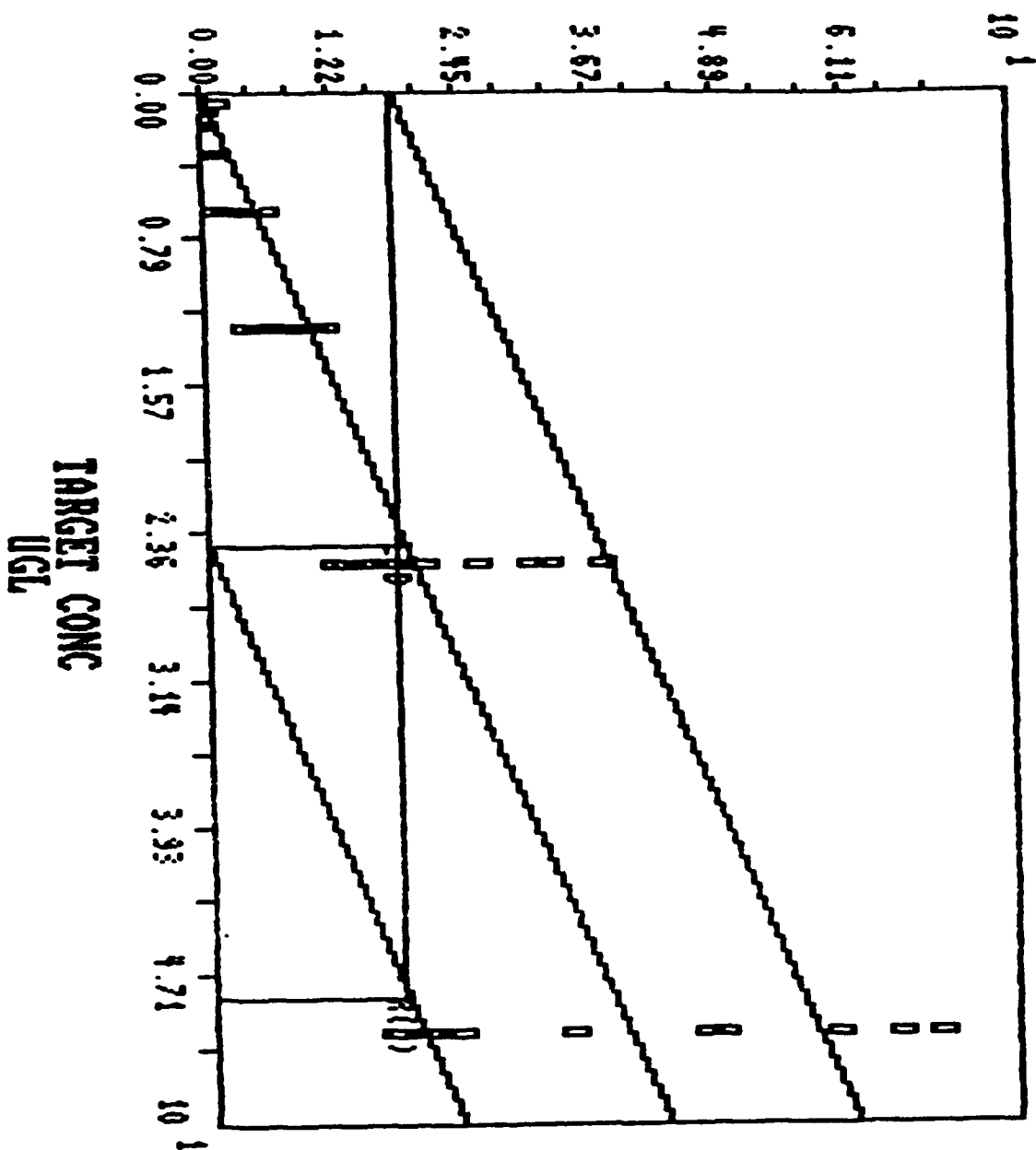


Figure F16
4-AM

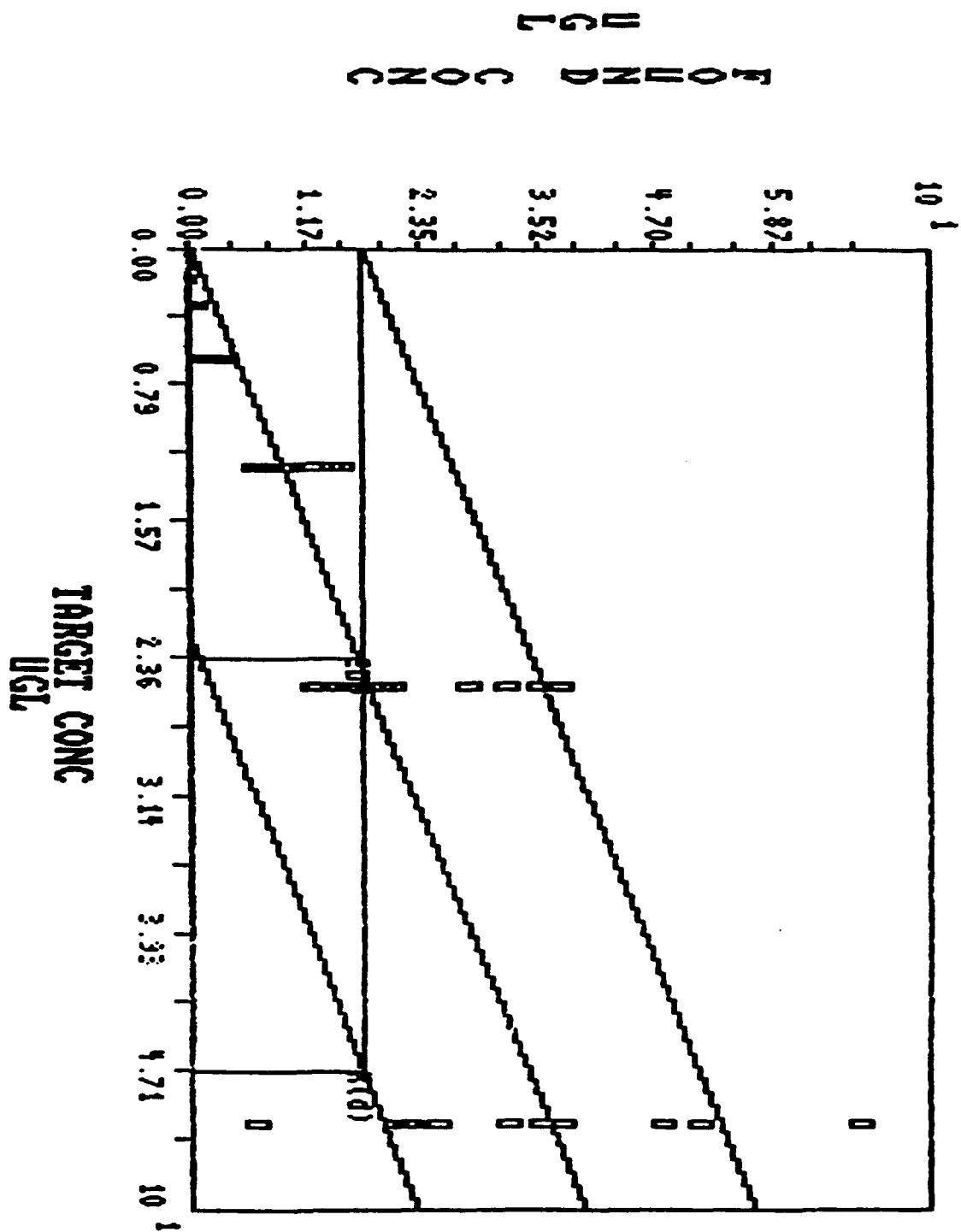


TABLE F9
CRITERION OF DETECTION FROM SOIL (mg/kg)

| <u>COMPOUNDS</u> | <u>CD</u> |
|------------------|-----------|
| HMX | 2.9 |
| TNB | 2.4 |
| RDX | 5.8 |
| TNT | 6.1 |
| 2,4 DNT | 5.7 |
| 2,6 DNT | 5.2 |
| 2-AM | 15.4 |
| 4-AM | 14.6 |

CERTIFICATION ANALYSIS

Table Fl0a

Report Date: 10/18/93

Method Name: HMX
Method Number:
Compound: HMX

Units of Measure: UGG
Laboratory: MA
Analysis Date: 01/23/91
Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
Y = (-0.00399784) + (1.017741420)X Y = (1.017141800)X

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 0.306303214 | 38 | 0.008060611 | 0.306750748 | 39 | 0.007865404 |
| Total Error: | 0.292517170 | 30 | 0.009750572 | 0.292517170 | 30 | 0.009750572 |
| Lack of Fit: | 0.013786044 | 8 | 0.001723255 | 0.014233578 | 9 | 0.001581509 |

LOF F-Ratio(F): 0.176733779 LOF F-Ratio(F): 0.162196496
Critical 95% F: 2.27 Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 0.055521102 Critical 95% F: 4.17

TABLE OF DATA POINTS

Targets: 10 Measures per Target: 4

| | Target Value | Found Concentration |
|-----|--------------|---|
| 1: | 10 | 9.8800000 9.9800000 10.410000 10.370000 |
| 2: | 5 | 4.9900000 5.0200000 5.2000000 5.2000000 |
| 3: | 2.5000000 | 2.5000000 2.5100000 2.5800000 2.5600000 |
| 4: | 1.2500000 | 1.2500000 1.4600000 1.2600000 1.3000000 |
| 5: | 0.6300000 | 0.6400000 0.6200000 0.6300000 0.6400000 |
| 6: | 0.3200000 | 0.3400000 0.3100000 0.2900000 0.2900000 |
| 7: | 0.1600000 | 0.1600000 0.1600000 0.1400000 0.1600000 |
| 8: | 0.0800000 | 0.0900000 0.0600000 0.0600000 0.0690000 |
| 9: | 0.0400000 | 0.0500000 0.0100000 0.0240000 0.0270000 |
| 10: | 0.0200000 | 0.0040000 0.0080000 0.0050000 0.0024000 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: RADFORD
 Method Number: 1
 Compound: HMX

Units of Measure: UGC
 Laboratory: MM
 Analysis Date: 12/31/91
 Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.00458677) + (1.017921390)X$ $Y = (1.017233440)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 0.308793193 | 38 | 0.008126137 | 0.309382294 | 39 | 0.007932879 |
| Total Error: | 0.294318503 | 30 | 0.009810617 | 0.294318503 | 30 | 0.009810617 |
| Lack of Fit: | 0.014474690 | 8 | 0.001809336 | 0.015063791 | 9 | 0.001673755 |

LOF F-Ratio(F): 0.184426351
 Critical 95% F: 2.27

LOF F-Ratio(F): 0.170606456
 Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 0.072494597 Critical 95% F: 4.17

TABLE OF DATA POINTS

Targets: 10

Measures per Target: 4

| | Target Value | Found Concentration |
|-----|--------------|---|
| 1: | 0.0200000 | 0.0040000 0.0080000 0.0050000 +2.40E-04 |
| 2: | 0.0400000 | 0.0500000 0.0100000 0.0240000 0.0270000 |
| 3: | 0.0800000 | 0.0900000 0.0600000 0.0600000 0.0690000 |
| 4: | 0.1600000 | 0.1600000 0.1600000 0.1400000 0.1600000 |
| 5: | 0.3200000 | 0.3400000 0.3100000 0.2900000 0.2900000 |
| 6: | 0.6300000 | 0.6400000 0.6200000 0.6300000 0.6400000 |
| 7: | 1.2500000 | 1.2500000 1.4600000 1.2600000 1.3000000 |
| 8: | 2.5000000 | 2.5800000 2.5800000 2.5000000 2.5100000 |
| 9: | 5 | 4.3900000 5.0200000 5.2000000 5.2000000 |
| 10: | 10 | 9.8800000 9.9800000 10.410000 10.370000 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Table F 11a

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: MILAN
 Method Number:
 Compound: TNB

Units of Measure: UGG
 Laboratory: MA
 Analysis Date: 01/23/91
 Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.04333250) + (1.013886250)X$ $Y = (1.007386980)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 0.293087156 | 38 | 0.007712820 | 0.345665012 | 39 | 0.008863205 |
| Total Error: | 0.217518860 | 30 | 0.007250629 | 0.217518860 | 30 | 0.007250629 |
| Lack of Fit: | 0.075568296 | 8 | 0.009446037 | 0.128146152 | 9 | 0.014238461 |

LOF F-Ratio(F): 1.302788687
 Critical 95% F: 2.27

LOF F-Ratio(F): 1.963755419
 Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

***Zero Intercept Rejected Calculated F: 6.816943312 Critical 95% F: 4.17

TABLE OF DATA POINTS

Targets: 10 Measures per Target: 4

| | Target Value | Found Concentration |
|-----|--------------|---|
| 1: | 10 | 10.310000 10.360000 9.9700000 9.9700000 |
| 2: | 5 | 4.8600000 4.9300000 5.0100000 5 |
| 3: | 2.5000000 | 2.4200000 2.3200000 2.4900000 2.4800000 |
| 4: | 1.2500000 | 1.0400000 1.2400000 1.2400000 1.2600000 |
| 5: | 0.6300000 | 0.5900000 0.5800000 0.6100000 0.6300000 |
| 6: | 0.3200000 | 0.2100000 0.1900000 0.3200000 0.3100000 |
| 7: | 0.1600000 | 0.1600000 0.1500000 0.1600000 0.1600000 |
| 8: | 0.0800000 | 0.0420000 0.0350000 0.0740000 0.0860000 |
| 9: | 0.0400000 | 0.0500000 0.0100000 0.0240000 0.0270000 |
| 10: | 0.0200000 | 0.0092000 0.0074000 0.0180000 0.0250000 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS

Table F 11b

Report Date: 10/12/93

Method Name: RADFORD
Method Number: 1
Compound: TNB

Units of Measure: UGG
Laboratory: MM
Analysis Date: 12/31/91
Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
Y = (-0.04162067) + (1.014855330)X Y = (1.008612820)X

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 0.257655922 | 38 | 0.006780419 | 0.306161703 | 39 | 0.007850300 |
| Total Error: | 0.204409860 | 30 | 0.006813662 | 0.204409860 | 30 | 0.006813662 |
| Lack of Fit: | 0.053246062 | 8 | 0.006655758 | 0.101751843 | 9 | 0.011305760 |

LOF F-Ratio(F): 0.976825347 LOF F-Ratio(F): 1.659278129
Critical 95% F: 2.27 Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

***Zero Intercept Rejected Calculated F: 7.153802884 Critical 95% F: 4.17

TABLE OF DATA POINTS

Targets: 10 Measures per Target: 4

| | Target Value | Found Concentration |
|-----|--------------|---|
| 1: | 0.0200000 | 0.0092000 0.0074000 0.0180000 0.0250000 |
| 2: | 0.0400000 | 0.0410000 0.0370000 0.0200000 0.0240000 |
| 3: | 0.0800000 | 0.0860000 0.0740000 0.0350000 0.0420000 |
| 4: | 0.1600000 | 0.0860000 0.0890000 0.1600000 0.1600000 |
| 5: | 0.3200000 | 0.2100000 0.1900000 0.3200000 0.3100000 |
| 6: | 0.6300000 | 0.6300000 0.6100000 0.5800000 0.5900000 |
| 7: | 1.2500000 | 1.0400000 1.2400000 1.2400000 1.2600000 |
| 8: | 2.5000000 | 2.4800000 2.4900000 2.5100000 2.5000000 |
| 9: | 5 | 4.8600000 4.9300000 5.0100000 5 |
| 10: | 10 | 9.9700000 9.9700000 10.3100000 10.3600000 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS

Table F 12a

Report Date: 10/18/93

Method Name: RDX
Method Number:
Compound: RDX

Units of Measure: UGG
Laboratory: MA
Analysis Date: 01/23/91
Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
Y = (-0.01057644) + (1.008113220)X Y = (1.006526900)X

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 0.059129398 | 38 | 0.001556037 | 0.062261634 | 39 | 0.001596452 |
| Total Error: | 0.035166500 | 30 | 0.001172217 | 0.035166500 | 30 | 0.001172217 |
| Lack of Fit: | 0.023962899 | 8 | 0.002995362 | 0.027095134 | 9 | 0.003010570 |

LOF F-Ratio(F): 2.555297495 LOF F-Ratio(F): 2.568271338
Critical 95% F: 2.27 Critical 95% F: 2.21
Data Not Linear Data Not Linear

ZERO INTERCEPT HYPOTHESIS

** Models not linear. Do not test Zero Intercept hypothesis.

Diagnose and correct analytical system before continuing.

TABLE OF DATA POINTS

Targets: 10 Measures per Target: 4

| | Target Value | Found Concentration |
|-----|--------------|---|
| 1: | 10 | 10.060000 10.150000 10.150000 10.060000 |
| 2: | 5 | 4.8900000 4.9400000 5.0500000 5.0200000 |
| 3: | 2.5000000 | 2.4400000 2.4700000 2.5100000 2.5200000 |
| 4: | 1.2500000 | 1.2100000 1.2300000 1.2200000 1.2900000 |
| 5: | 0.6300000 | 0.6300000 0.6100000 0.6200000 0.6200000 |
| 6: | 0.3200000 | 0.3400000 0.3300000 0.3400000 0.3100000 |
| 7: | 0.1600000 | 0.1600000 0.1500000 0.1700000 0.1900000 |
| 8: | 0.0800000 | 0.0790000 0.0900000 0.0880000 0.1000000 |
| 9: | 0.0400000 | 0.0230000 0.0310000 0.0310000 0.0500000 |
| 10: | 0.0200000 | 0.0320000 0.0200000 0.0020000 0.0020000 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS

Report Date: 10/19/93

Method Name: RDX
 Method Number:
 Compound: RDX

Units of Measure: UGG
 Laboratory: MA
 Analysis Date: 01/23/91
 Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.01050523) + (1.008102610)X$ $Y = (1.006526980)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 0.059094913 | 38 | 0.001555129 | 0.062185112 | 39 | 0.001594490 |
| Total Error: | 0.035115500 | 30 | 0.001170517 | 0.035115500 | 30 | 0.001170517 |
| Lack of Fit: | 0.023979413 | 8 | 0.002997427 | 0.027069612 | 9 | 0.003007735 |

LOF F-Ratio(F): 2.560772294 LOF F-Ratio(F): 2.569578676
 Critical 95% F: 2.27 Critical 95% F: 2.21
 Data Not Linear Data Not Linear

ZERO INTERCEPT HYPOTHESIS

** Models not linear. Do not test Zero Intercept hypothesis.

Diagnose and correct analytical system before continuing.

TABLE OF DATA POINTS

Targets: 10

Measures per Target: 4

| | Target Value | Found Concentration |
|-----|--------------|---|
| 1: | 10 | 10.060000 10.150000 10.150000 10.060000 |
| 2: | 5 | 4.8900000 4.9400000 5.0500000 5.0200000 |
| 3: | 2.5000000 | 2.4400000 2.4700000 2.5100000 2.5200000 |
| 4: | 1.2500000 | 1.2100000 1.2300000 1.2200000 1.2900000 |
| 5: | 0.6300000 | 0.6300000 0.6100000 0.6200000 0.6200000 |
| 6: | 0.3200000 | 0.3400000 0.3300000 0.3400000 0.3100000 |
| 7: | 0.1600000 | 0.1600000 0.1500000 0.1700000 0.1900000 |
| 8: | 0.0800000 | 0.0790000 0.0900000 0.0880000 0.1000000 |
| 9: | 0.0400000 | 0.0230000 0.0310000 0.0310000 0.0500000 |
| 10: | 0.0200000 | 0.0320000 0.0200000 0.0020000 0.0020000 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS

Table F 12b

Report Date: 10/12/93

Method Name: RADFORD
Method Number: 1
Compound: RDX

Units of Measure: UGG
Laboratory: MM
Analysis Date: 12/31/91
Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
Y = (0.013858142) + (1.001916230)X Y = (1.003992260)X

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 0.373006260 | 34 | 0.010970772 | 0.377621272 | 35 | 0.010789179 |
| Total Error: | 0.226222000 | 27 | 0.008378593 | 0.226222000 | 27 | 0.008378593 |
| Lack of Fit: | 0.146784260 | 7 | 0.020969180 | 0.151399272 | 8 | 0.018924909 |

LOF F-Ratio(F): 2.502709109 LOF F-Ratio(F): 2.258721711
Critical 95% F: 2.37 Critical 95% F: 2.31
Data Not Linear

ZERO INTERCEPT HYPOTHESIS

** Intercept model not linear. Do not test Zero Intercept hypothesis.

Diagnose and correct analytical system before continuing.

TABLE OF DATA POINTS

Targets: 9

Measures per Target: 4

| | Target Value | Found Concentration | | Target Value | Found Concentration |
|----|--------------|---------------------|-----------|--------------|---------------------|
| 1: | 0.0400000 | 0 | 0 | 0.0270000 | 0.0270000 |
| 2: | 0.0800000 | 0 | 0.0580000 | 0.0600000 | 0.0600000 |
| 3: | 0.1600000 | 0.1400000 | 0.2100000 | 0.1900000 | 0.1900000 |
| 4: | 0.3200000 | 0.2600000 | 0.3900000 | 0.1900000 | 0.3400000 |
| 5: | 0.6250000 | 0.6100000 | 0.6300000 | 0.5800000 | 0.5800000 |
| 6: | 1.2500000 | 1.5000000 | 1.4000000 | 1.3000000 | 1.1000000 |
| 7: | 2.5000000 | 2.6000000 | 2.5000000 | 2.8000000 | 2.8000000 |
| 8: | 5 | 5.1000000 | 5.1000000 | 4.9000000 | 4.9000000 |
| 9: | 10 | 10 | 10.010000 | 10 | 10 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Table F 13a

CERTIFICATION ANALYSIS

Report Date: 10/19/93

Method Name: TNT
 Method Number:
 Compound: TNT

Units of Measure: UGG
 Laboratory: MA
 Analysis Date: 01/23/91
 Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.01813630) + (1.007155650)X$ $Y = (1.004435460)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 0.113801306 | 38 | 0.002994771 | 0.123011588 | 39 | 0.003154143 |
| Total Error: | 0.102973750 | 30 | 0.003432458 | 0.102973750 | 30 | 0.003432458 |
| Lack of Fit: | 0.010827556 | 8 | 0.001353444 | 0.020037838 | 9 | 0.002226426 |

LOF F-Ratio(F): 0.394307627 LOF F-Ratio(F): 0.648639030
 Critical 95% F: 2.27 Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 3.075454301 Critical 95% F: 4.17

TABLE OF DATA POINTS

Targets: 10

Measures per Target: 4

| | Target Value | Found Concentration |
|-----|--------------|---|
| 1: | 10 | 9.9300000 10.110000 10.180000 10.080000 |
| 2: | 5 | 4.8600000 4.8900000 5.1100000 5.0700000 |
| 3: | 2.5000000 | 2.4600000 2.4400000 2.5200000 2.5500000 |
| 4: | 1.2500000 | 1.1500000 1.2000000 1.2300000 1.2900000 |
| 5: | 0.6300000 | 0.6200000 0.5900000 0.6400000 0.6200000 |
| 6: | 0.3200000 | 0.3200000 0.2900000 0.3100000 0.3400000 |
| 7: | 0.1600000 | 0.1400000 0.1400000 0.1600000 0.1800000 |
| 8: | 0.0800000 | 0.0640000 0.0660000 0.0740000 0.0840000 |
| 9: | 0.0400000 | 0.0280000 0.0280000 0.0270000 0.0260000 |
| 10: | 0.0200000 | 0.0020000 0.0020000 0.0140000 0.0120000 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Table F 13b

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: RADFORD
 Method Number: 1
 Compound: TNT

Units of Measure: UGG
 Laboratory: MM
 Analysis Date: 12/31/91
 Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.01801080) + (1.007417900)X$ $Y = (1.004716530)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 0.110208263 | 38 | 0.002900217 | 0.119291521 | 39 | 0.003058757 |
| Total Error: | 0.101346750 | 30 | 0.003378225 | 0.101346750 | 30 | 0.003378225 |
| Lack of Fit: | 0.008861513 | 8 | 0.001107689 | 0.017944771 | 9 | 0.001993863 |

LOF F-Ratio(F): 0.327890867
 Critical 95% F: 2.27

LOF F-Ratio(F): 0.590210375
 Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 3.131923094 Critical 95% F: 4.17

TABLE OF DATA POINTS

Targets: 10 Measures per Target: 4

| | Target Value | Found Concentration |
|-----|--------------|---|
| 1: | 0.0200000 | 0 0 0.0140000 0.0120000 |
| 2: | 0.0400000 | 0.0260000 0.0270000 0.0280000 0.0280000 |
| 3: | 0.0800000 | 0.0640000 0.0660000 0.0740000 0.0840000 |
| 4: | 0.1600000 | 0.1400000 0.1400000 0.1600000 0.1800000 |
| 5: | 0.3200000 | 0.3400000 0.3100000 0.3200000 0.2900000 |
| 6: | 0.6300000 | 0.6200000 0.5900000 0.6400000 0.6200000 |
| 7: | 1.2500000 | 1.2900000 1.2300000 1.1500000 1.2000000 |
| 8: | 2.5000000 | 2.4600000 2.4400000 2.5200000 2.5500000 |
| 9: | 5 | 5.0700000 5.1100000 4.8400000 4.9400000 |
| 10: | 10 | 9.9300000 10.1100000 10.1800000 10.0800000 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Table F 14a

CERTIFICATION ANALYSIS

Report Date: 10/18/93

Method Name: 2,4
 Method Number:
 Compound: 2,4

Units of Measure: UGG
 Laboratory: MA
 Analysis Date: 01/23/91
 Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.02530612) + (1.025863060)X$ $Y = (1.022067500)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 0.405071561 | 38 | 0.010659778 | 0.423003471 | 39 | 0.010846243 |
| Total Error: | 0.360487280 | 30 | 0.012016243 | 0.360487280 | 30 | 0.012016243 |
| Lack of Fit: | 0.044584281 | 8 | 0.005573035 | 0.062516191 | 9 | 0.006946243 |

LOF F-Ratio(F): 0.463791826
 Critical 95% F: 2.27

LOF F-Ratio(F): 0.578071169
 Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 1.682202963 Critical 95% F: 4.17

TABLE OF DATA POINTS

Targets: 10

Measures per Target: 4

| | Target Value | Found Concentration |
|-----|--------------|---|
| 1: | 10 | 10.090000 10.110000 10.560000 10.360000 |
| 2: | 5 | 4.8400000 4.8500000 5.2500000 5.2000000 |
| 3: | 2.5000000 | 2.3700000 2.3900000 2.5700000 2.6300000 |
| 4: | 1.2500000 | 1.2000000 1.2000000 1.2600000 1.3100000 |
| 5: | 0.6300000 | 0.6200000 0.5900000 0.6500000 0.6700000 |
| 6: | 0.3200000 | 0.3400000 0.3400000 0.3100000 0.3100000 |
| 7: | 0.1600000 | 0.1500000 0.1500000 0.1600000 0.1900000 |
| 8: | 0.0800000 | 0.0730000 0.0720000 0.0800000 0.0730000 |
| 9: | 0.0400000 | 0.0220000 0.0140000 0.0088000 0.0360000 |
| 10: | 0.0200000 | 0.0020000 0.0020000 0.0020000 0.0020000 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS

Table F 14b

Report Date: 10/12/93

Method Name: RADFORD
Method Number: 1
Compound: 2-4DNT

Units of Measure: UGG
Laboratory: MM
Analysis Date: 12/31/91
Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
Y = (-0.02459154) + (1.023768270)X Y = (1.020079880)X

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 0.460537936 | 38 | 0.012119419 | 0.477471436 | 39 | 0.012242857 |
| Total Error: | 0.416563030 | 30 | 0.013885434 | 0.416563030 | 30 | 0.013885434 |
| Lack of Fit: | 0.043974906 | 8 | 0.005496863 | 0.060908406 | 9 | 0.006767601 |

LOF F-Ratio(F): 0.395872619 LOF F-Ratio(F): 0.487388475
Critical 95% F: 2.27 Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 1.397220402 Critical 95% F: 4.17

TABLE OF DATA POINTS

Targets: 10

Measures per Target: 4

| | Target Value | Found Concentration |
|-----|--------------|---|
| 1: | 0.0200000 | 0 0 0 0 |
| 2: | 0.0400000 | 0.0220000 0.0140000 0.0088000 0.0360000 |
| 3: | 0.0800000 | 0.0730000 0.0800000 0.0720000 0.0730000 |
| 4: | 0.1600000 | 0.1500000 0.1500000 0.1600000 0.1900000 |
| 5: | 0.3200000 | 0.3100000 0.3100000 0.3400000 0.3400000 |
| 6: | 0.6300000 | 0.6200000 0.5900000 0.6500000 0.6700000 |
| 7: | 1.2500000 | 1.2000000 1.2000000 1.2600000 1.3100000 |
| 8: | 2.5000000 | 2.3700000 2.3900000 2.5700000 2.6300000 |
| 9: | 5 | 4.8400000 4.8000000 5.2500000 5.2000000 |
| 10: | 10 | 10.009000 10.110000 10.560000 10.360000 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Table F 15a

CERTIFICATION ANALYSIS

Report Date: 10/18/93

Method Name: 2,6
 Method Number:
 Compound: 2,6

Units of Measure: UGG
 Laboratory: MA
 Analysis Date: 01/23/91
 Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.03122974) + (1.047214870)X$ $Y = (1.042530850)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 1.981234090 | 38 | 0.052137739 | 2.008543500 | 39 | 0.051501115 |
| Total Error: | 1.940400000 | 30 | 0.064680000 | 1.940400000 | 30 | 0.064680000 |
| Lack of Fit: | 0.040834090 | 8 | 0.005104261 | 0.068143500 | 9 | 0.007571500 |

LOF F-Ratio(F): 0.078915604 LOF F-Ratio(F): 0.117060915
 Critical 95% F: 2.27 Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 0.523793521 Critical 95% F: 4.17

TABLE OF DATA POINTS

Targets: 10 Measures per Target: 4

| | Target Value | Found Concentration | | | |
|-----|--------------|---------------------|----------|-----------|-----------|
| 1: | 10 | 10.140000 | 9.810000 | 11.240000 | 10.730000 |
| 2: | 5 | 4.780000 | 4.800000 | 5.570000 | 5.460000 |
| 3: | 2.500000 | 2.320000 | 2.320000 | 2.680000 | 2.770000 |
| 4: | 1.250000 | 1.380000 | 1.290000 | 1.260000 | 1.210000 |
| 5: | 0.630000 | 0.600000 | 0.590000 | 0.680000 | 0.710000 |
| 6: | 0.320000 | 0.320000 | 0.280000 | 0.350000 | 0.370000 |
| 7: | 0.160000 | 0.170000 | 0.210000 | 0.140000 | 0.120000 |
| 8: | 0.080000 | 0.059000 | 0.046000 | 0.080000 | 0.043000 |
| 9: | 0.020000 | 0 | 0 | 0 | 0 |
| 10: | 0.040000 | 0 | 0 | 0 | 0 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Table F 15b

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: RADFORD
 Method Number: 1
 Compound: 2-6DNT

Units of Measure: UGG
 Laboratory: MM
 Analysis Date: 12/31/91
 Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.03122974) + (1.047214870)X$ $Y = (1.042530850)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 1.981234090 | 38 | 0.052137739 | 2.008543500 | 39 | 0.051501115 |
| Total Error: | 1.940400000 | 30 | 0.064680000 | 1.940400000 | 30 | 0.064680000 |
| Lack of Fit: | 0.040834090 | 8 | 0.005104261 | 0.068143500 | 9 | 0.007571500 |

LOF F-Ratio(F): 0.078915604 LOF F-Ratio(F): 0.117060915
 Critical 95% F: 2.27 Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

Zero Intercept Accepted Calculated F: 0.523793521 Critical 95% F: 4.17

TABLE OF DATA POINTS

Targets: 10 Measures per Target: 4

| | Target Value | Found Concentration | | | |
|-----|--------------|---------------------|-----------|-----------|-----------|
| 1: | 0.0200000 | 0 | 0 | 0 | 0 |
| 2: | 0.0400000 | 0 | 0 | 0 | 0 |
| 3: | 0.0800000 | 0.0460000 | 0.0590000 | 0.0800000 | 0.0430000 |
| 4: | 0.1600000 | 0.1200000 | 0.1400000 | 0.1700000 | 0.2100000 |
| 5: | 0.3200000 | 0.3200000 | 0.2800000 | 0.3500000 | 0.3700000 |
| 6: | 0.6300000 | 0.7100000 | 0.6800000 | 0.5900000 | 0.6000000 |
| 7: | 1.2500000 | 1.2600000 | 1.2100000 | 1.2900000 | 1.3800000 |
| 8: | 2.5000000 | 2.7700000 | 2.6800000 | 2.3200000 | 2.3200000 |
| 9: | 5 | 4.7800000 | 4.8000000 | 5.5700000 | 5.4600000 |
| 10: | 10 | 10.140000 | 9.8100000 | 11.240000 | 10.730000 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS

Report Date: 10/18/93

Method Name: 2AM
 Method Number:
 Compound: 2AM

Units of Measure: UGG
 Laboratory: MA
 Analysis Date 01/23/91
 Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.04092383) + (1.009736910)X$ $Y = (1.003598910)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 0.263036377 | 38 | 0.006922010 | 0.309931526 | 39 | 0.007946962 |
| Total Error: | 0.218409500 | 30 | 0.007280317 | 0.218409500 | 30 | 0.007280317 |
| Lack of Fit: | 0.044626877 | 8 | 0.005578360 | 0.091522026 | 9 | 0.010169114 |

LOF F-Ratio(F): 0.766224861 LOF F-Ratio(F): 1.396795561
 Critical 95% F: 2.27 Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

***Zero Intercept Rejected Calculated F: 6.774787892 Critical 95% F: 4.17

TABLE OF DATA POINTS

Targets: 10

Measures per Target: 4

| | Target Value | Found Concentration |
|-----|--------------|---|
| 1: | 10 | 9.9800000 10.030000 10.430000 9.9500000 |
| 2: | 5 | 4.8300000 5.0500000 4.9200000 5.0400000 |
| 3: | 2.5000000 | 2.3800000 2.4100000 2.4900000 2.4100000 |
| 4: | 1.2500000 | 1.1900000 1.1700000 1.2100000 1.1700000 |
| 5: | 0.6300000 | 0.6200000 0.5700000 0.5600000 0.7100000 |
| 6: | 0.3200000 | 0.2500000 0.3300000 0.3400000 0.3300000 |
| 7: | 0.1600000 | 0.1400000 0.1800000 0.1400000 0.0750000 |
| 8: | 0.0800000 | 0.0430000 0.0800000 0.0230000 0.0560000 |
| 9: | 0.0400000 | 0.0190000 0.0020000 0.0040000 0.0020000 |
| 10: | 0.0200000 | 0.0020000 0.0020000 0.0020000 0.0020000 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Table F 16b

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: RADFORD
 Method Number: 1
 Compound: 2AMDNT

Units of Measure: UGG
 Laboratory: MM
 Analysis Date: 12/31/91
 Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.04248105) + (1.009965530)X$ $Y = (1.003593960)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 0.262130576 | 38 | 0.006898173 | 0.312662515 | 39 | 0.008016988 |
| Total Error: | 0.218400500 | 30 | 0.007280017 | 0.218400500 | 30 | 0.007280017 |
| Lack of Fit: | 0.043730076 | 8 | 0.005466260 | 0.094262015 | 9 | 0.010473557 |

LOF F-Ratio(F): 0.750858102 LOF F-Ratio(F): 1.438672149
 Critical 95% F: 2.27 Critical 95% F: 2.21

ZERO INTERCEPT HYPOTHESIS

 **Zero Intercept Rejected Calculated F: 7.325409005 Critical 95% F: 4.17

TABLE OF DATA POINTS

Targets: 10 Measures per Target: 4

| | Target Value | Found Concentration |
|-----|--------------|---------------------|
| 1: | 0.0200000 | 0.0200000 |
| 2: | 0.0400000 | 0.0400000 |
| 3: | 0.0800000 | 0.0800000 |
| 4: | 0.1600000 | 0.1600000 |
| 5: | 0.3200000 | 0.3200000 |
| 6: | 0.6300000 | 0.6300000 |
| 7: | 1.2500000 | 1.2500000 |
| 8: | 2.5000000 | 2.5000000 |
| 9: | 5 | 5 |
| 10: | 10 | 10 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

Table F 17a

CERTIFICATION ANALYSIS

Report Date: 10/18/93

Method Name: MILAN
 Method Number:
 Compound: 4AMDNT

Units of Measure: UGG
 Laboratory: MA
 Analysis Date: 01/23/91
 Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.05365346) + (1.006851730)X$ $Y = (0.998804462)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 0.181320988 | 38 | 0.004771605 | 0.261927629 | 39 | 0.006716093 |
| Total Error: | 0.138595000 | 30 | 0.004619833 | 0.138595000 | 30 | 0.004619833 |
| Lack of Fit: | 0.042725988 | 8 | 0.005340748 | 0.123332629 | 9 | 0.013703625 |

LOF F-Ratio(F): 1.156047873
 Critical 95% F: 2.27

LOF F-Ratio(F): 2.966259702
 Critical 95% F: 2.21
 Data Not Linear

ZERO INTERCEPT HYPOTHESIS

 Zero Intercept Rejected Calculated F: 16.89298295 Critical 95% F: 4.17
 Model not linear through Origin

TABLE OF DATA POINTS

Targets: 10

Measures per Target: 4

| | Target Value | Found Concentration |
|-----|--------------|---|
| 1: | 10 | 10.030000 10 10.160000 9.9900000 |
| 2: | 5 | 4.8700000 4.8500000 4.9100000 5.1000000 |
| 3: | 2.5000000 | 2.3800000 2.4000000 2.4900000 2.4900000 |
| 4: | 1.2500000 | 1.2100000 1.1600000 1.2200000 1.2100000 |
| 5: | 0.6300000 | 0.6000000 0.6100000 0.5800000 0.6500000 |
| 6: | 0.3200000 | 0.3500000 0.3200000 0.2100000 0.0360000 |
| 7: | 0.1600000 | 0.0600000 0.0650000 0.1100000 0.0810000 |
| 8: | 0.0800000 | 0.0210000 0.0320000 0.0360000 0.0210000 |
| 9: | 0.0400000 | 0.0830000 0.0360000 0.0210000 0.0020000 |
| 10: | 0.0200000 | 0.0020000 0.0020000 0.0020000 0.0020000 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

CERTIFICATION ANALYSIS

Report Date: 10/12/93

Method Name: RADFORD
 Method Number: 1
 Compound: 4AMDNT

Units of Measure: UGG
 Laboratory: MM
 Analysis Date: 12/31/91
 Matrix: WA

ANALYSIS OF RESIDUAL VARIATIONS

--- Model with Intercept --- - Model through the Origin -
 $Y = (-0.05243419) + (1.006758340)X$ $Y = (0.998893951)X$

| | (SS) | (df) | (MS) | (SS) | (df) | (MS) |
|--------------|-------------|------|-------------|-------------|------|-------------|
| Residual: | 0.134476662 | 38 | 0.003538860 | 0.211461379 | 39 | 0.005422087 |
| Total Error: | 0.106517568 | 30 | 0.003550586 | 0.106517568 | 30 | 0.003550586 |
| Lack of Fit: | 0.027959094 | 8 | 0.003494887 | 0.104943811 | 9 | 0.011660423 |

LOF F-Ratio(F): 0.984312771 LOF F-Ratio(F): 3.284084587
 Critical 95% F: 2.27 Critical 95% F: 2.21
 Data Not Linear

ZERO INTERCEPT HYPOTHESIS

 Zero Intercept Rejected Calculated F: 21.75410367 Critical 95% F: 4.17
 Model not linear through origin

TABLE OF DATA POINTS

Targets: 10 Measures per Target: 4

| | Target Value | Found Concentration |
|-----|--------------|--|
| 1: | 0.0200000 | 0 0 0 0 |
| 2: | 0.0400000 | 0.0083000 0 0 0.0190000 |
| 3: | 0.0800000 | 0.0210000 0.0360000 0.0320000 0.0210000 |
| 4: | 0.1600000 | 0.0650000 0.0600000 0.1100000 0.0810000 |
| 5: | 0.3200000 | 0.1200000 0.2900000 0.3200000 0.3500000 |
| 6: | 0.6300000 | 0.6100000 0.6000000 0.5800000 0.6500000 |
| 7: | 1.2500000 | 1.2200000 1.2100000 1.1600000 1.2100000 |
| 8: | 2.5000000 | 2.3800000 2.4000000 2.4900000 2.4900000 |
| 9: | 5 | 4.8700000 4.8500000 5.1000000 4.9100000 |
| 10: | 10 | 10.030000 10 10.160000 9.9900000 |

*** END OF CERTIFICATION LACK OF FIT DATA TABLE ***

FIGURE F ^{17a}
HMX

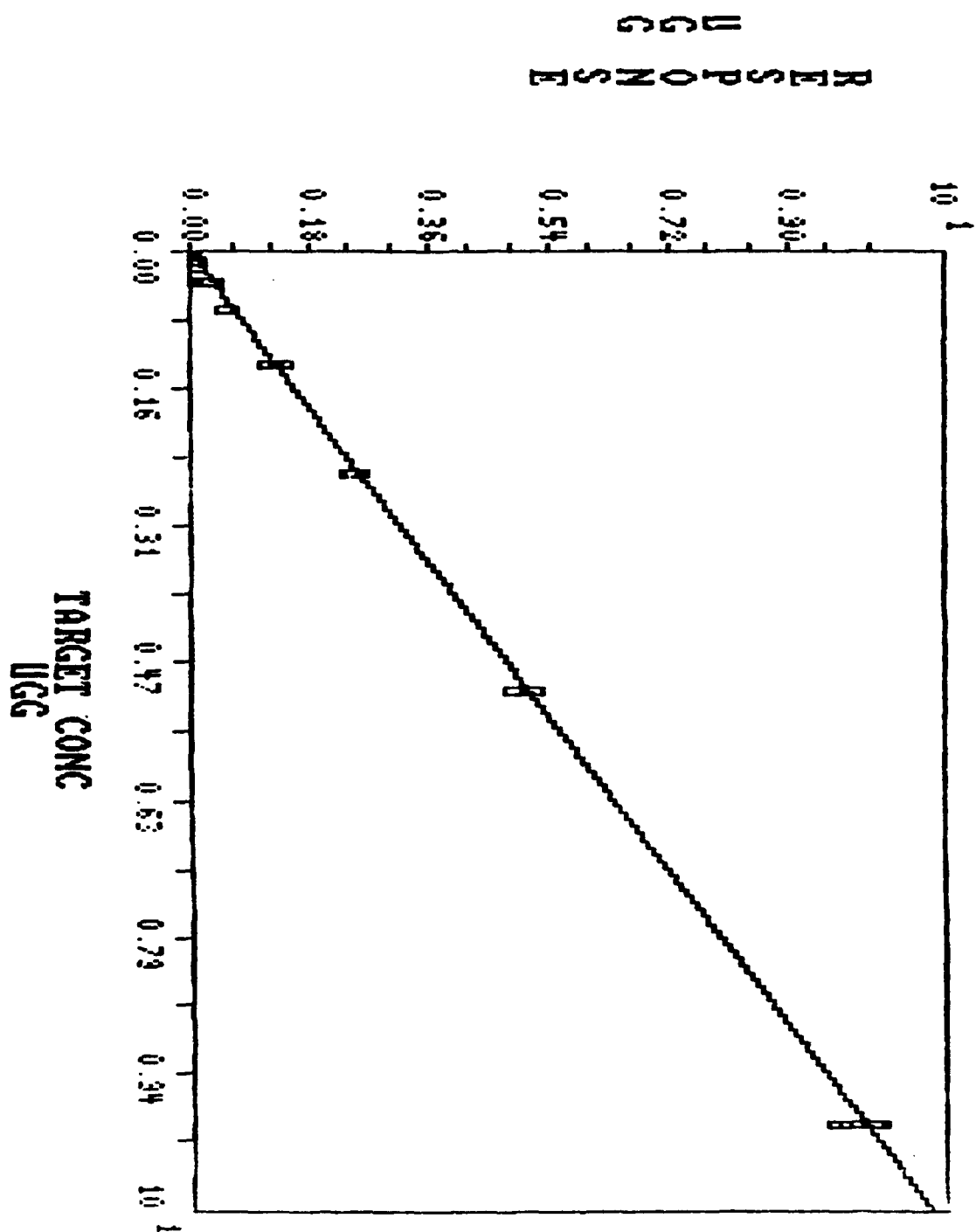


FIGURE F 17b

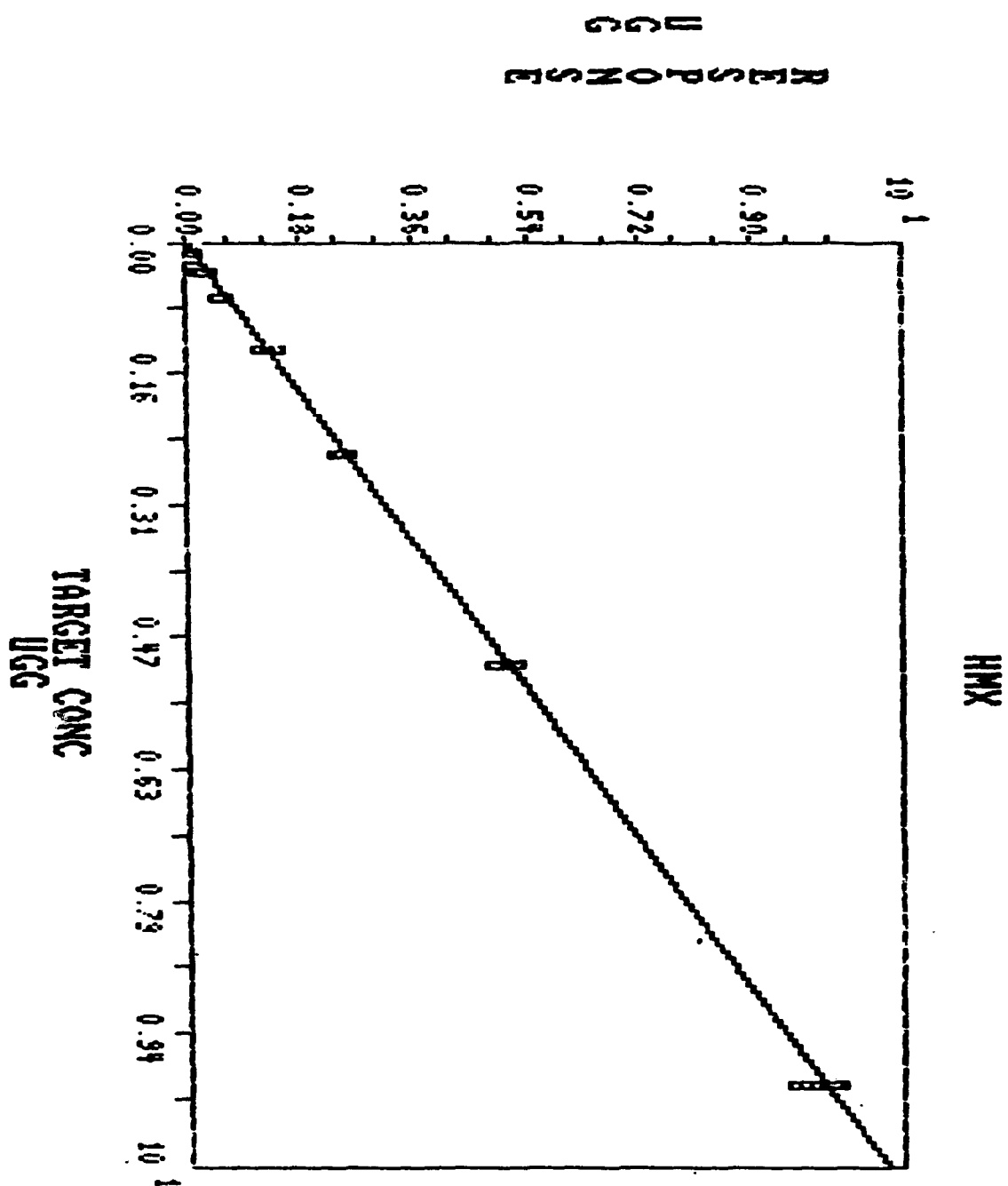


FIGURE F 18a

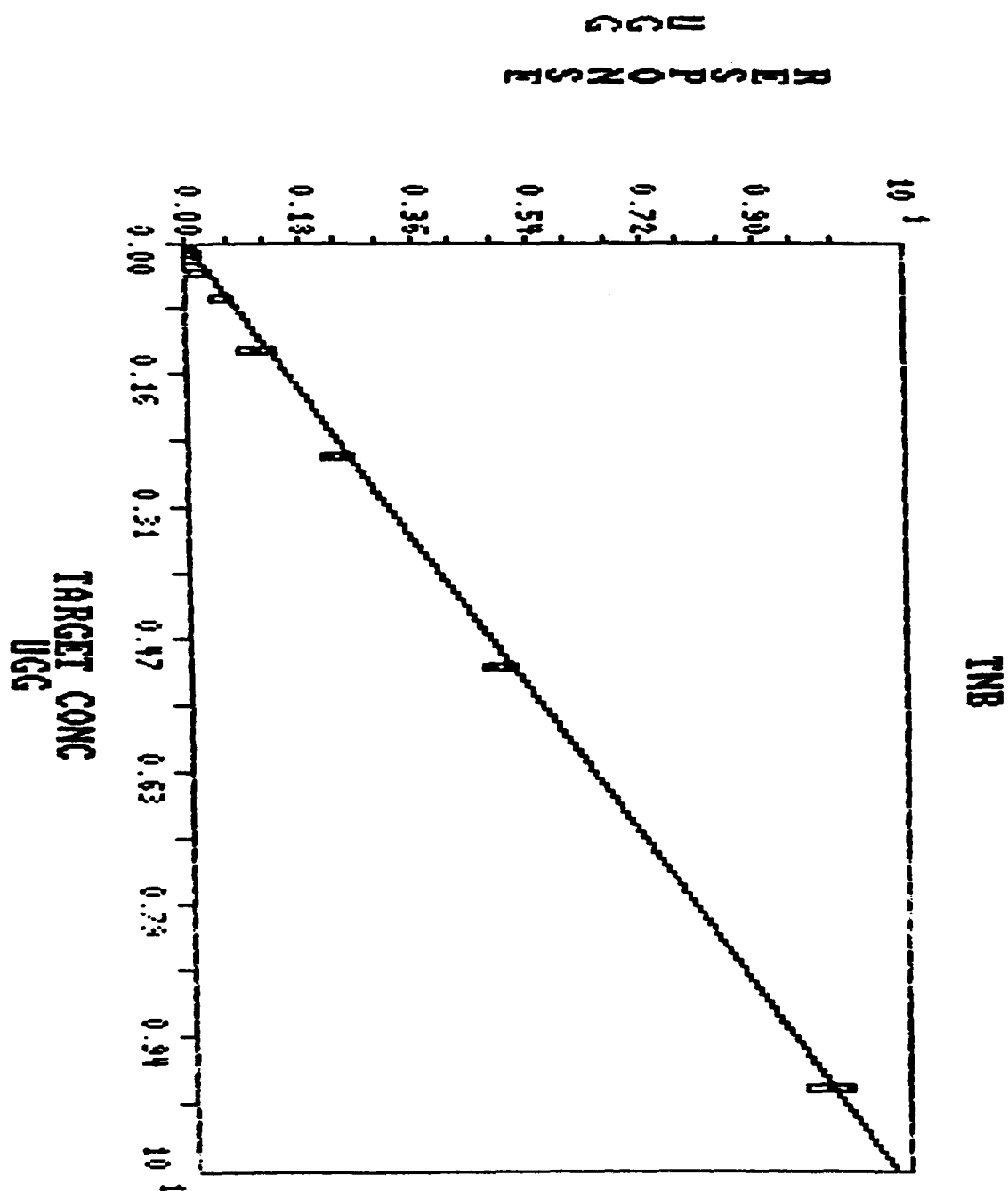


FIGURE F 18b

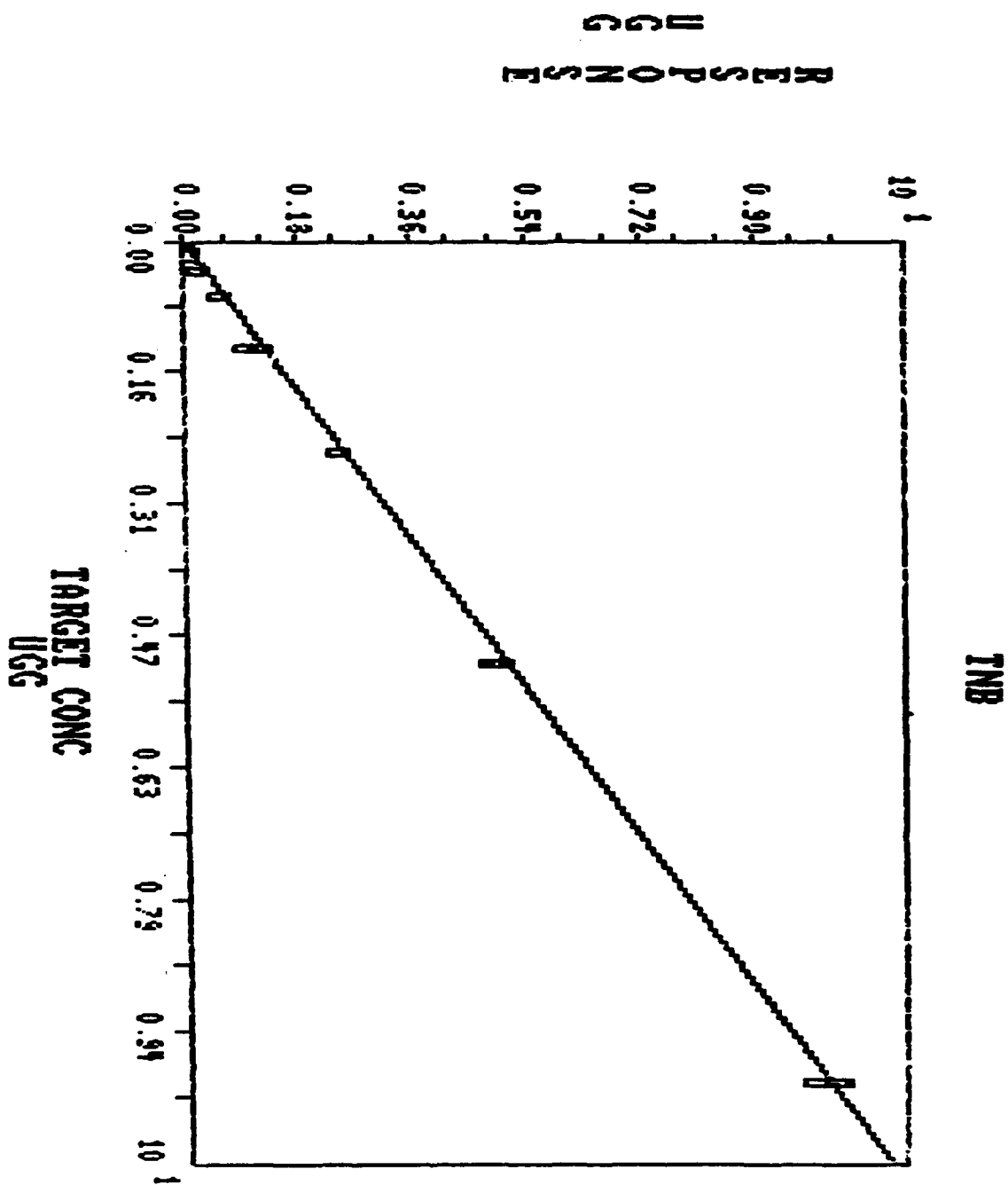


FIGURE F 19a
RDX

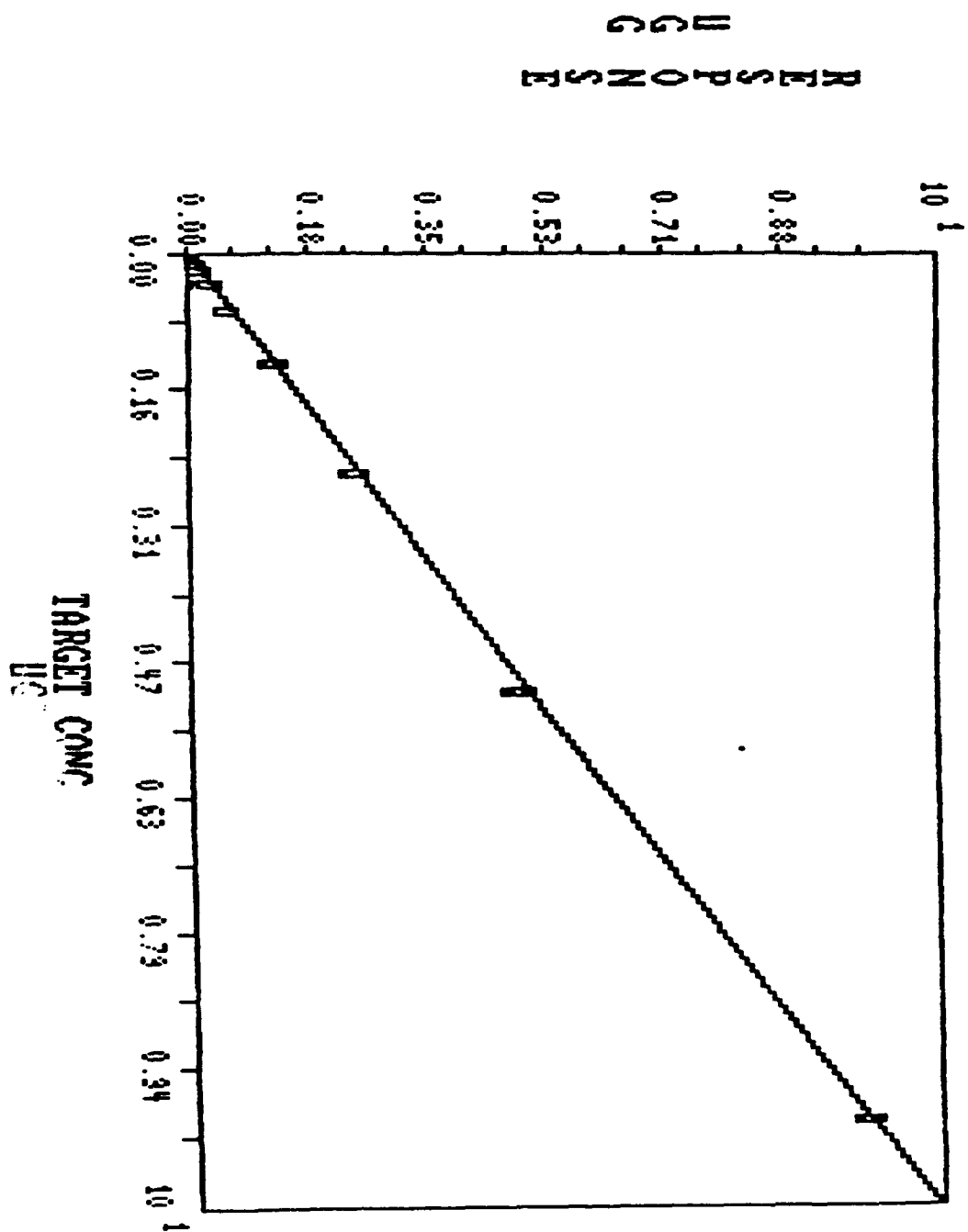


FIGURE F 19b

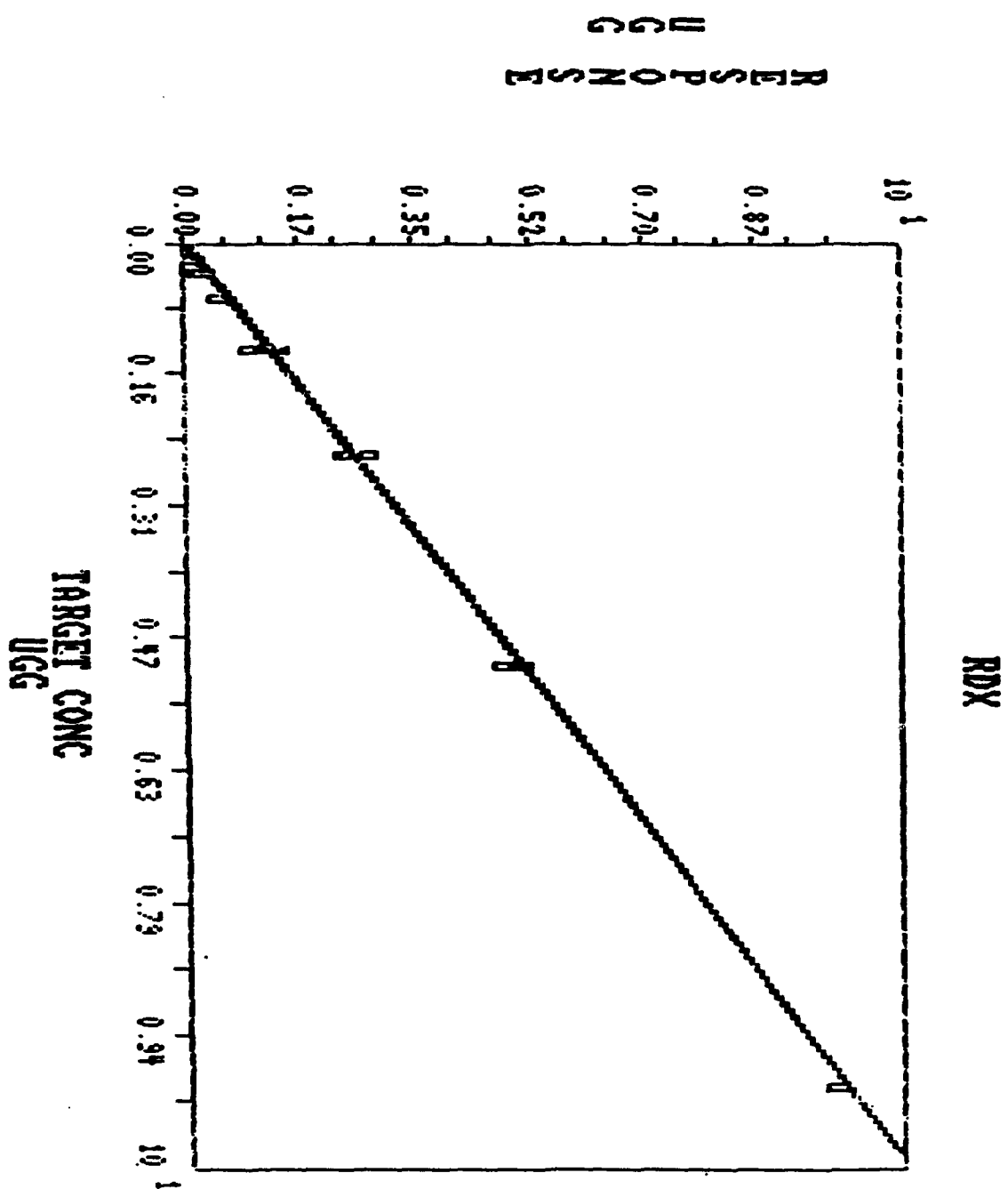


FIGURE F 20a

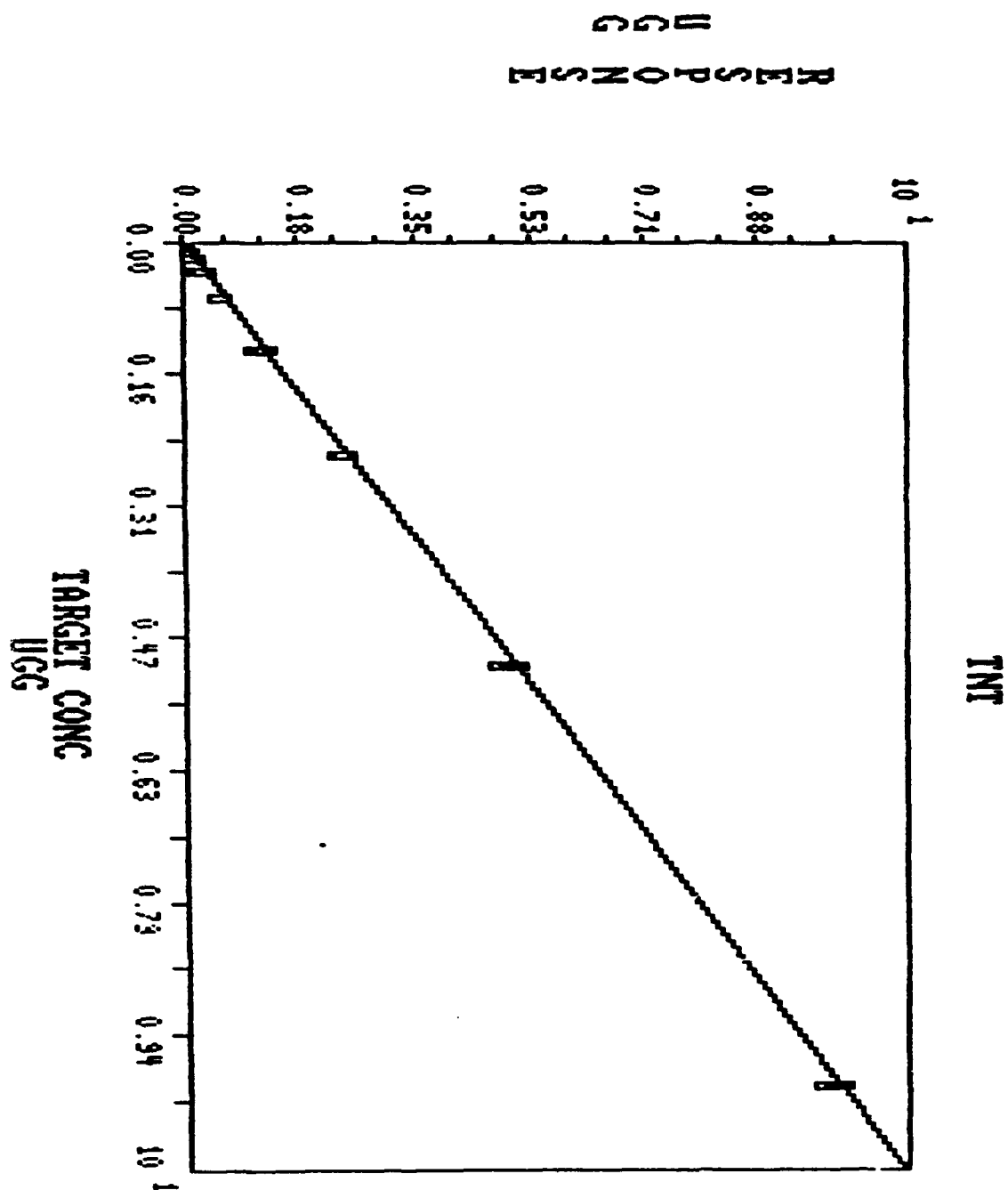


FIGURE F 20b

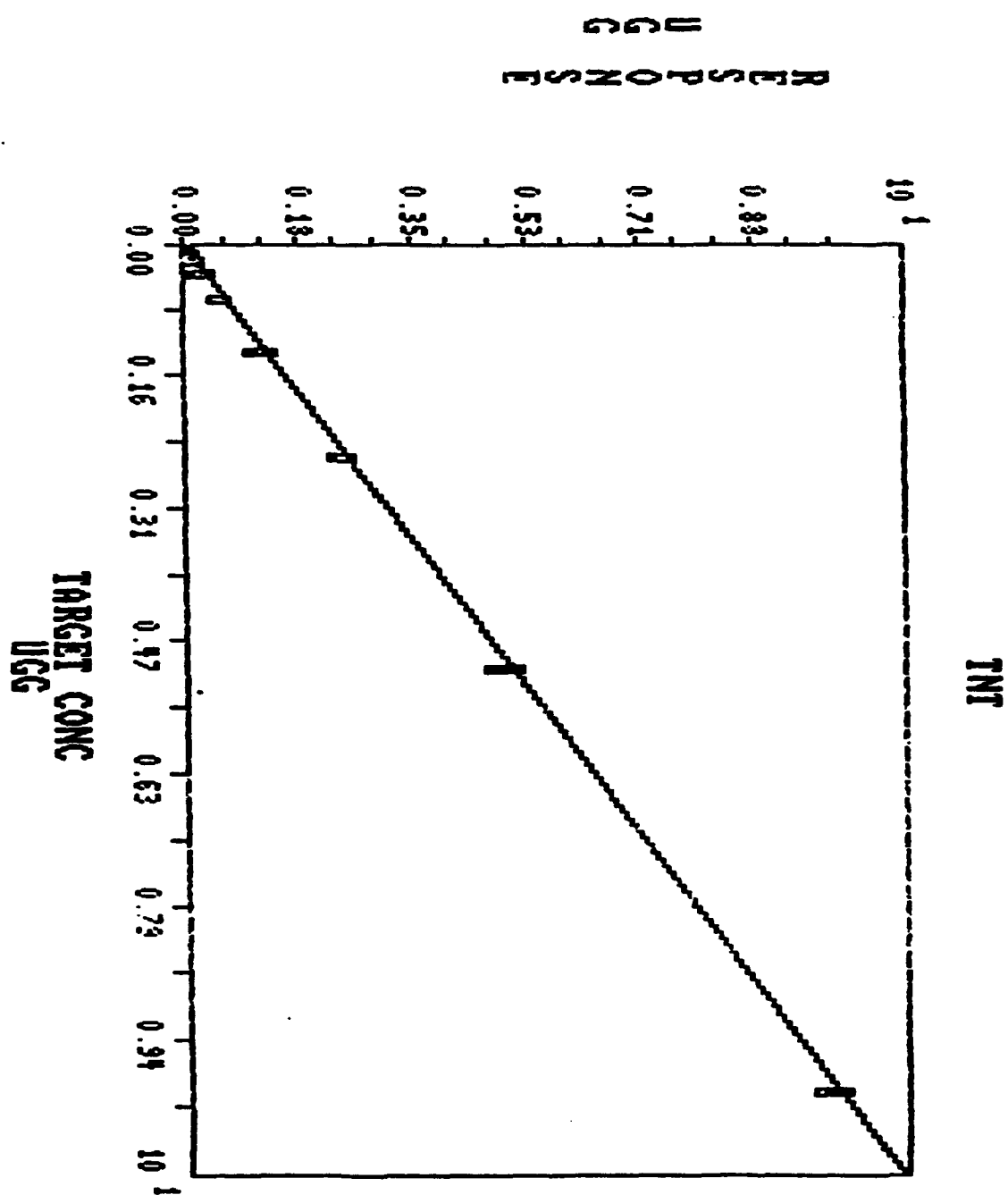


FIGURE F 21a
2,4

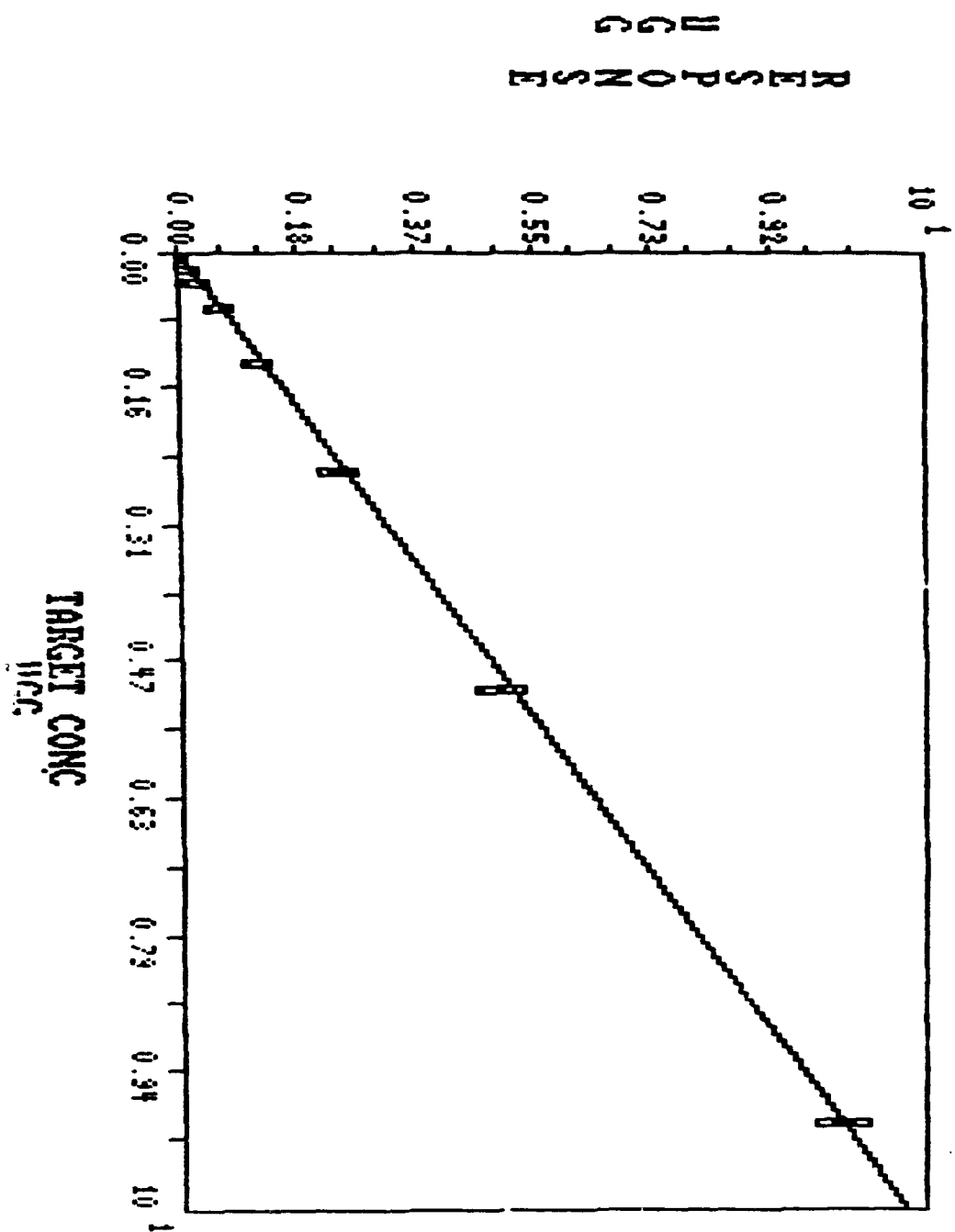


FIGURE F 21b

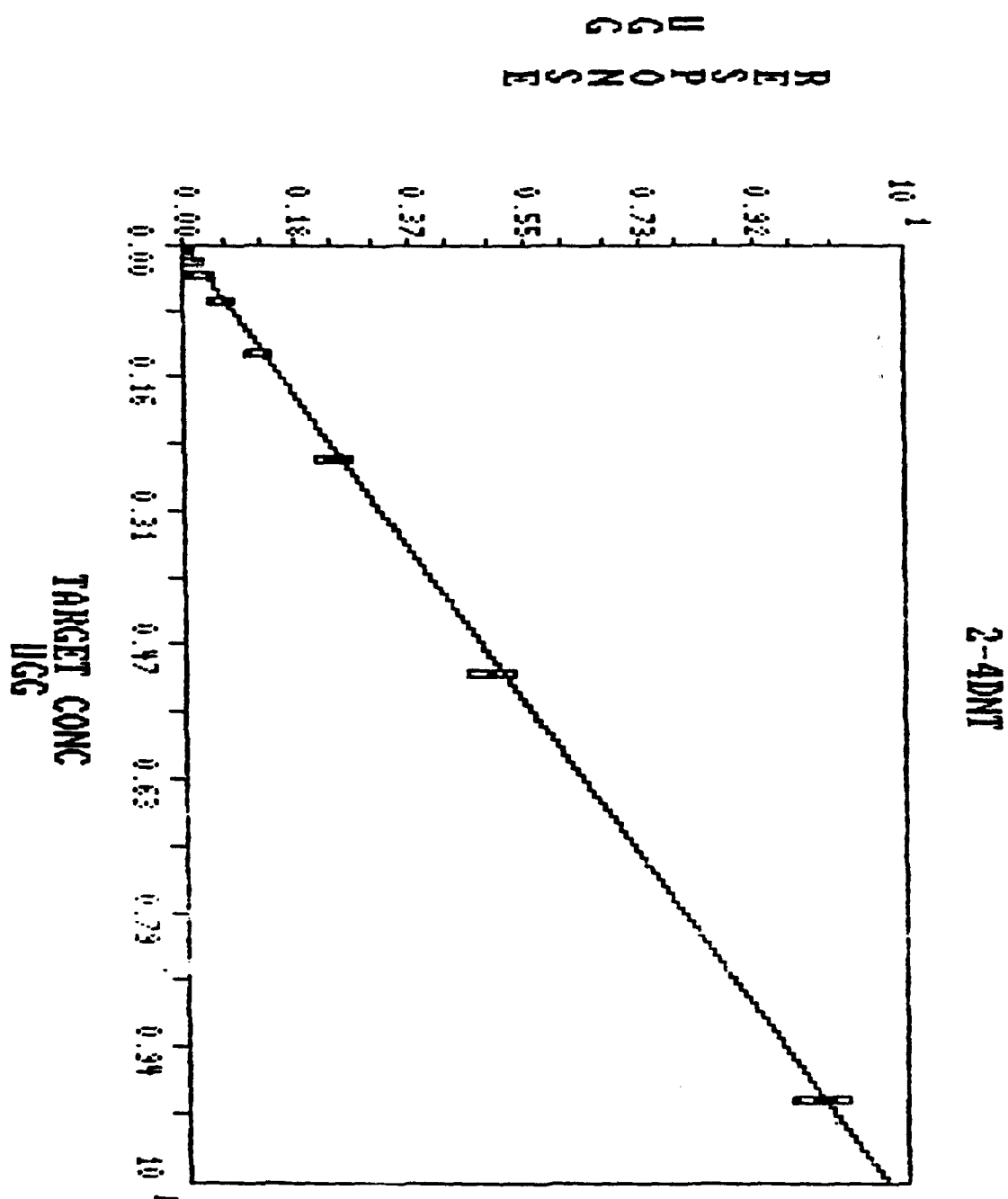


FIGURE F 22a

2,6

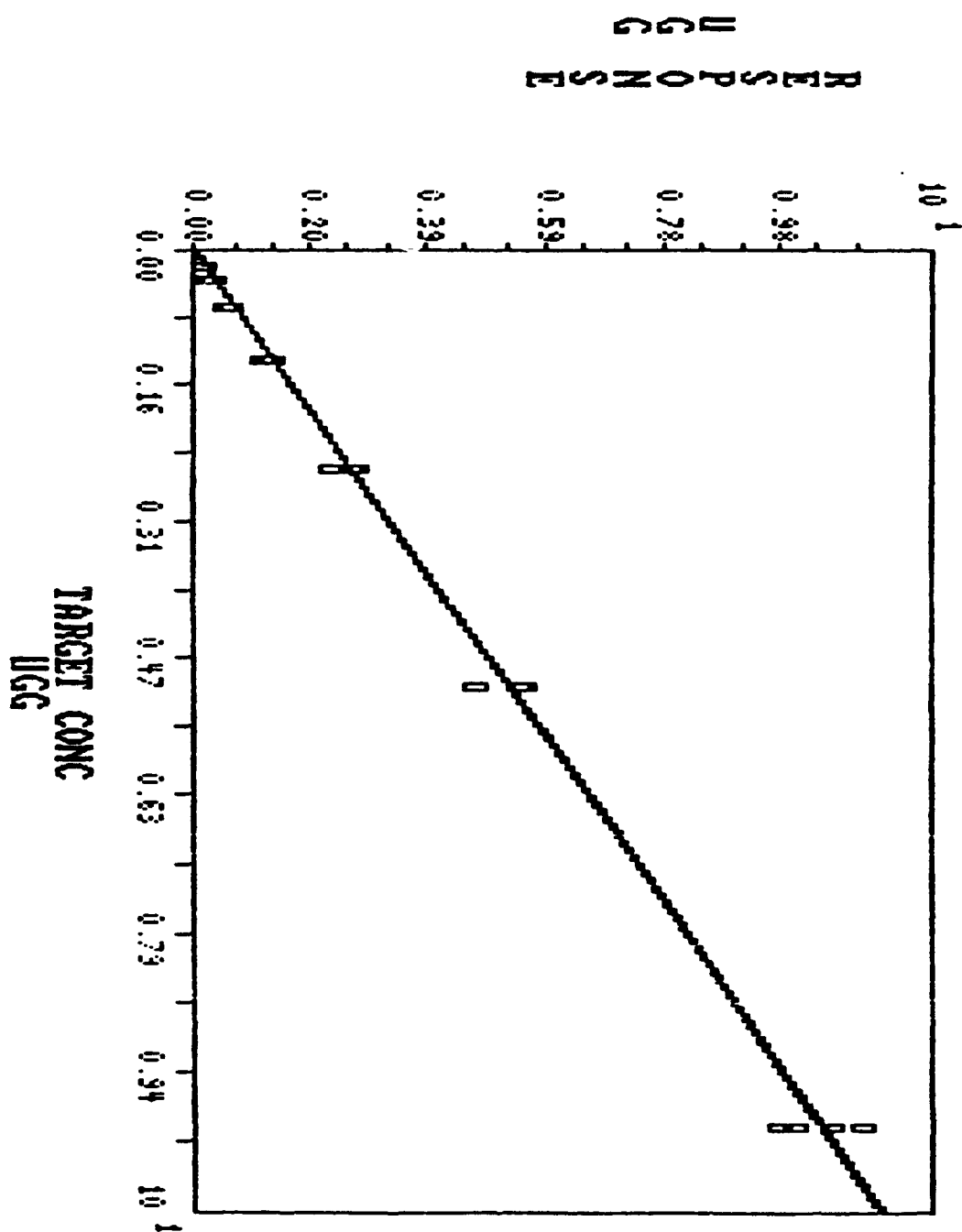


FIGURE F 22b

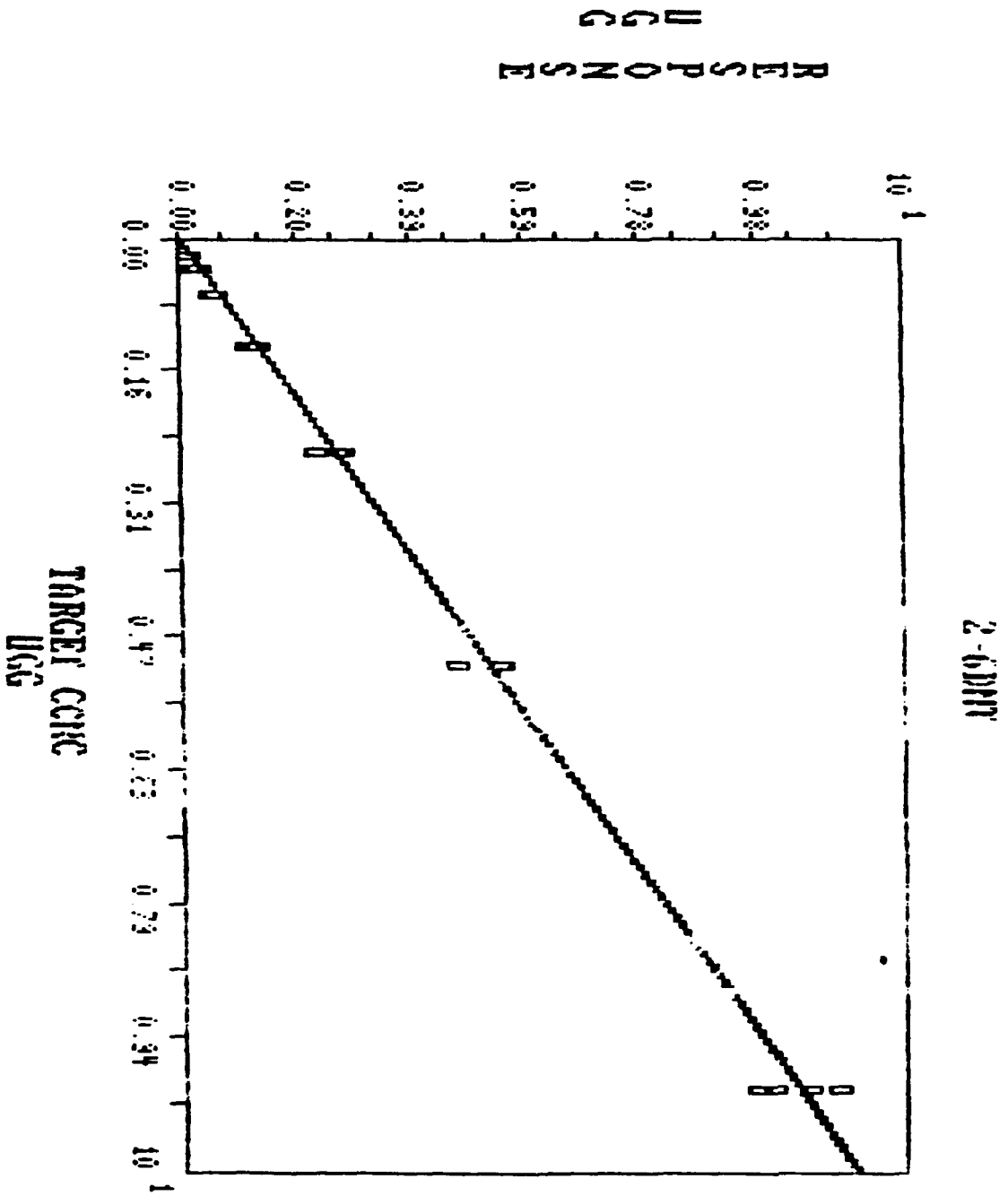


FIGURE F 23a

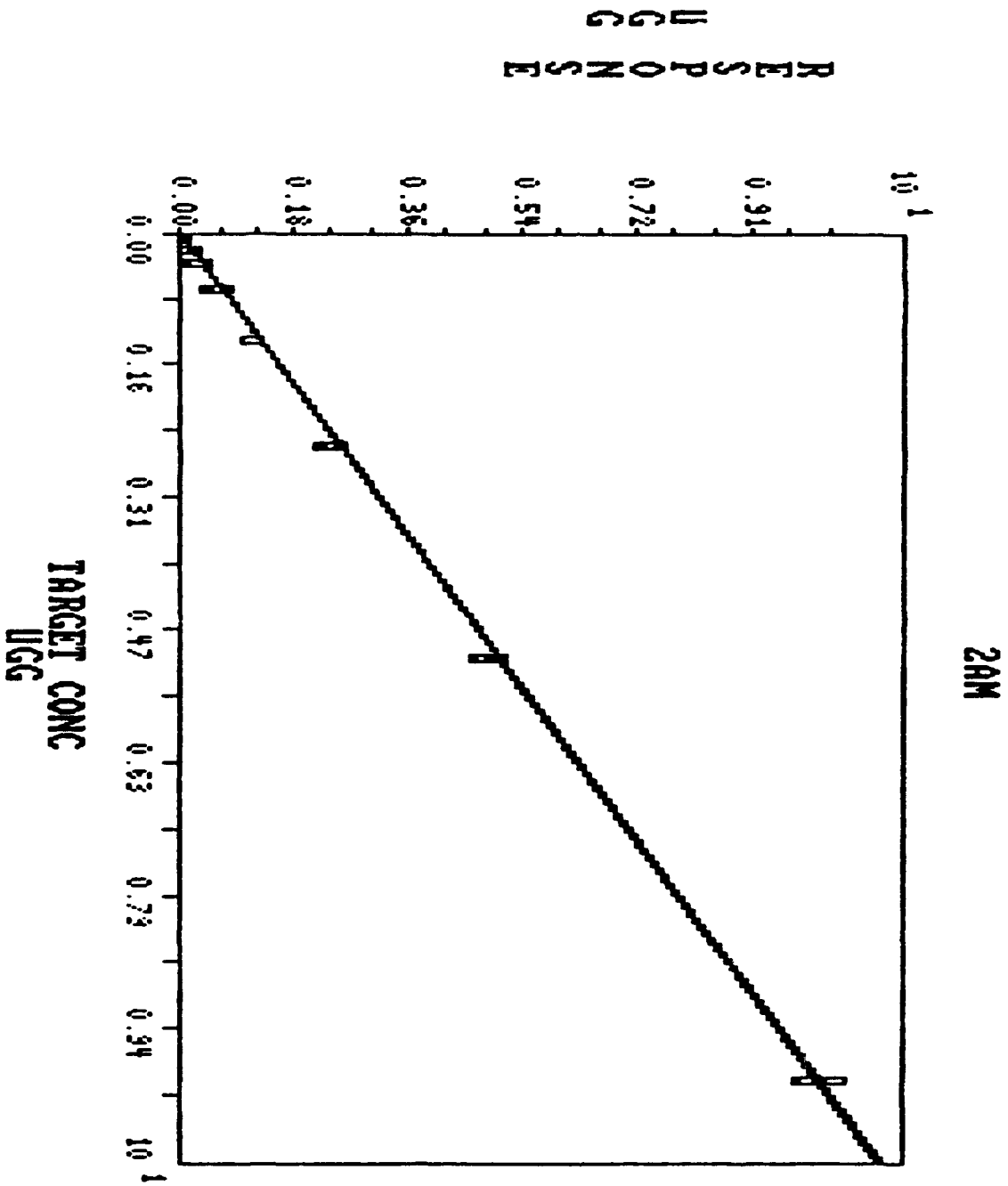


FIGURE F 23b

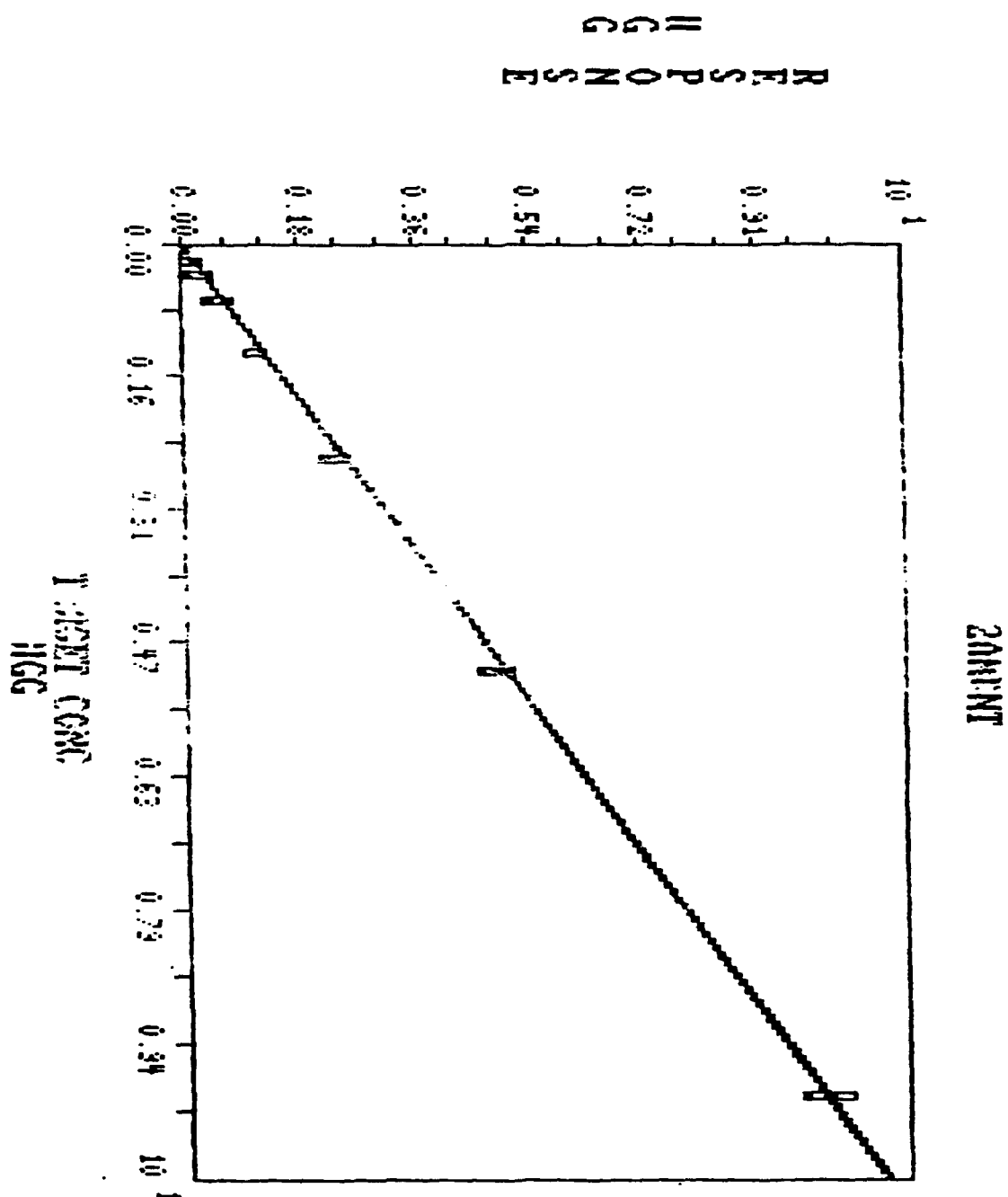


FIGURE F 24a

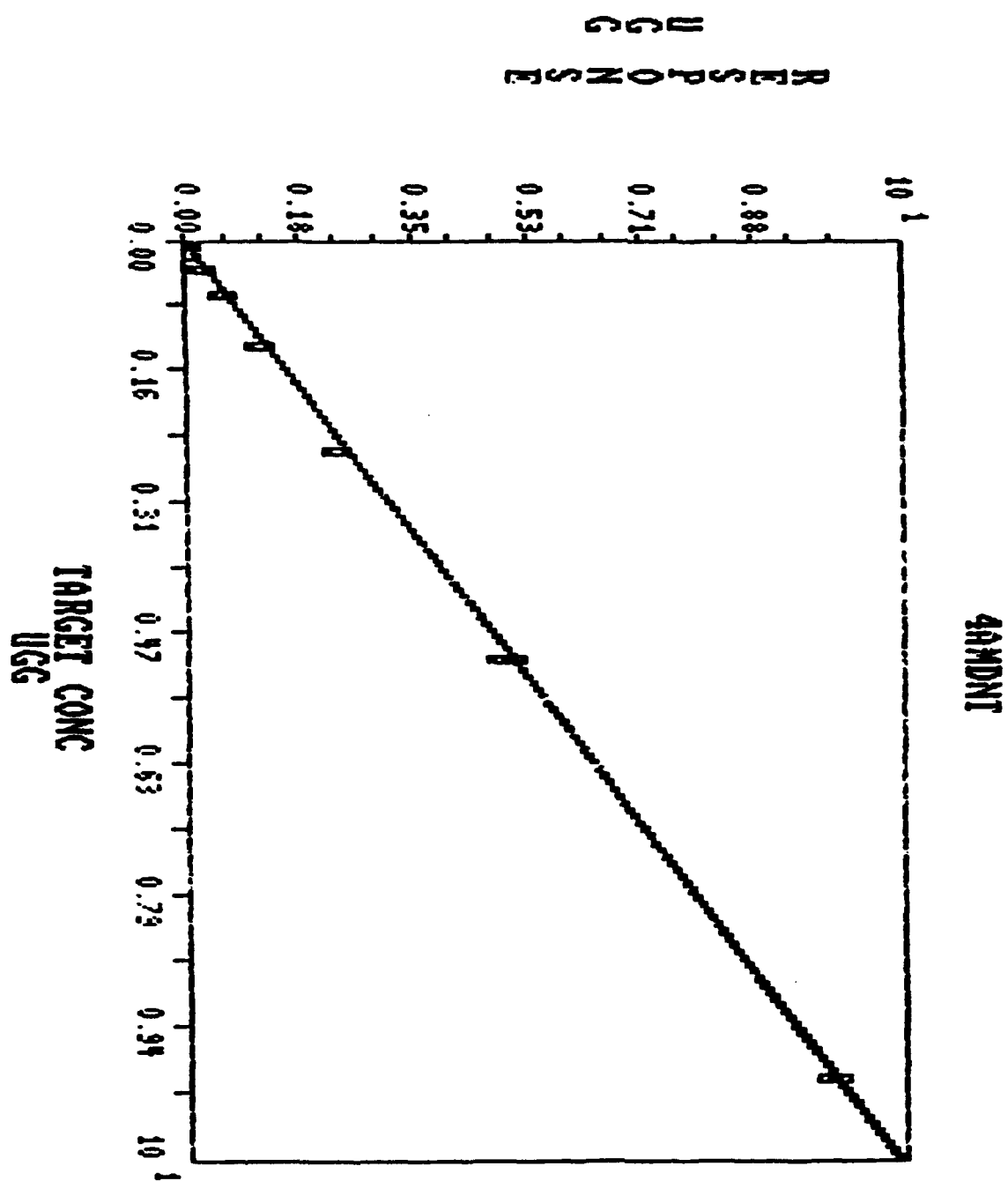


FIGURE F 24b

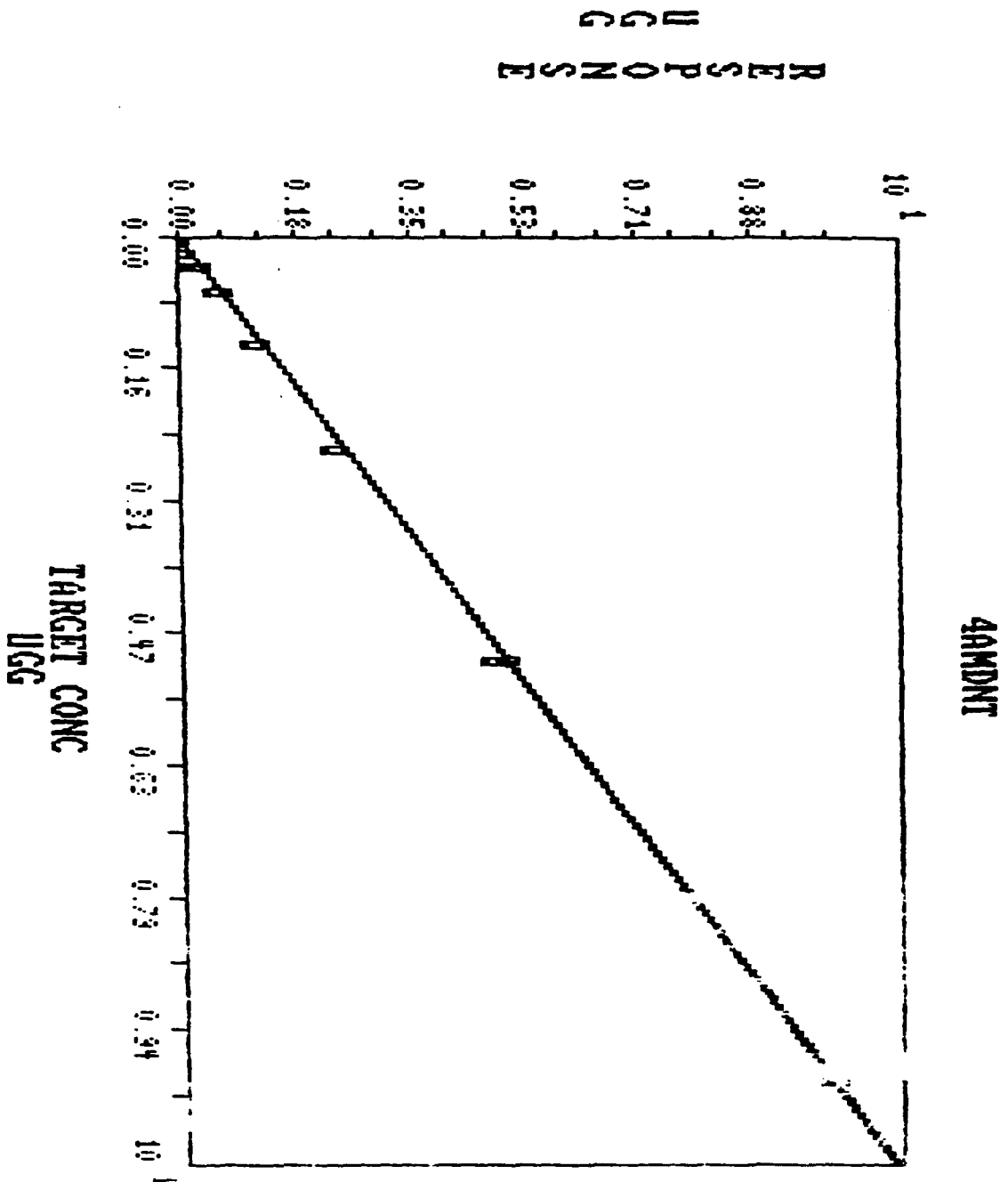


Figure F 25a

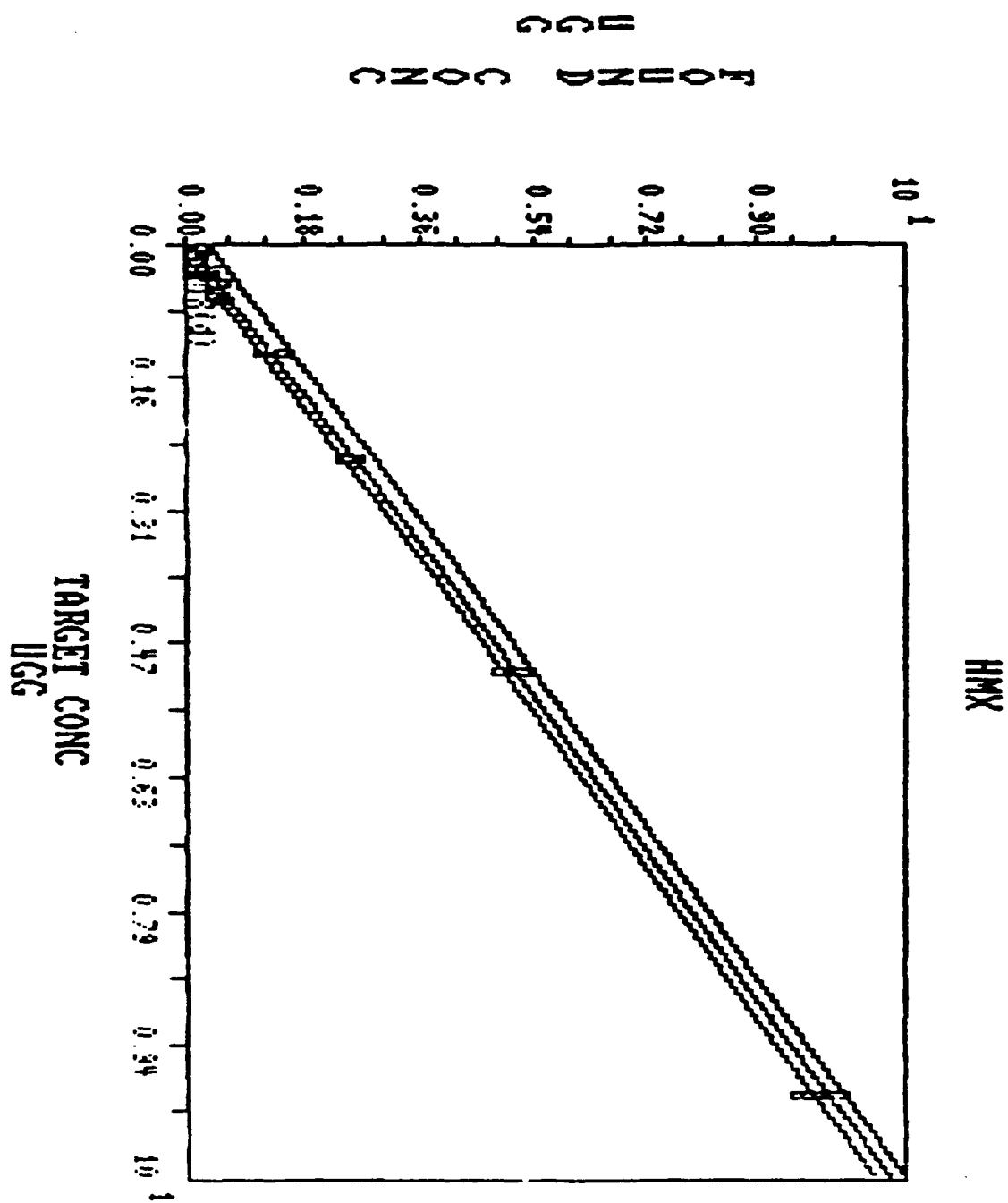


FIGURE F 25b

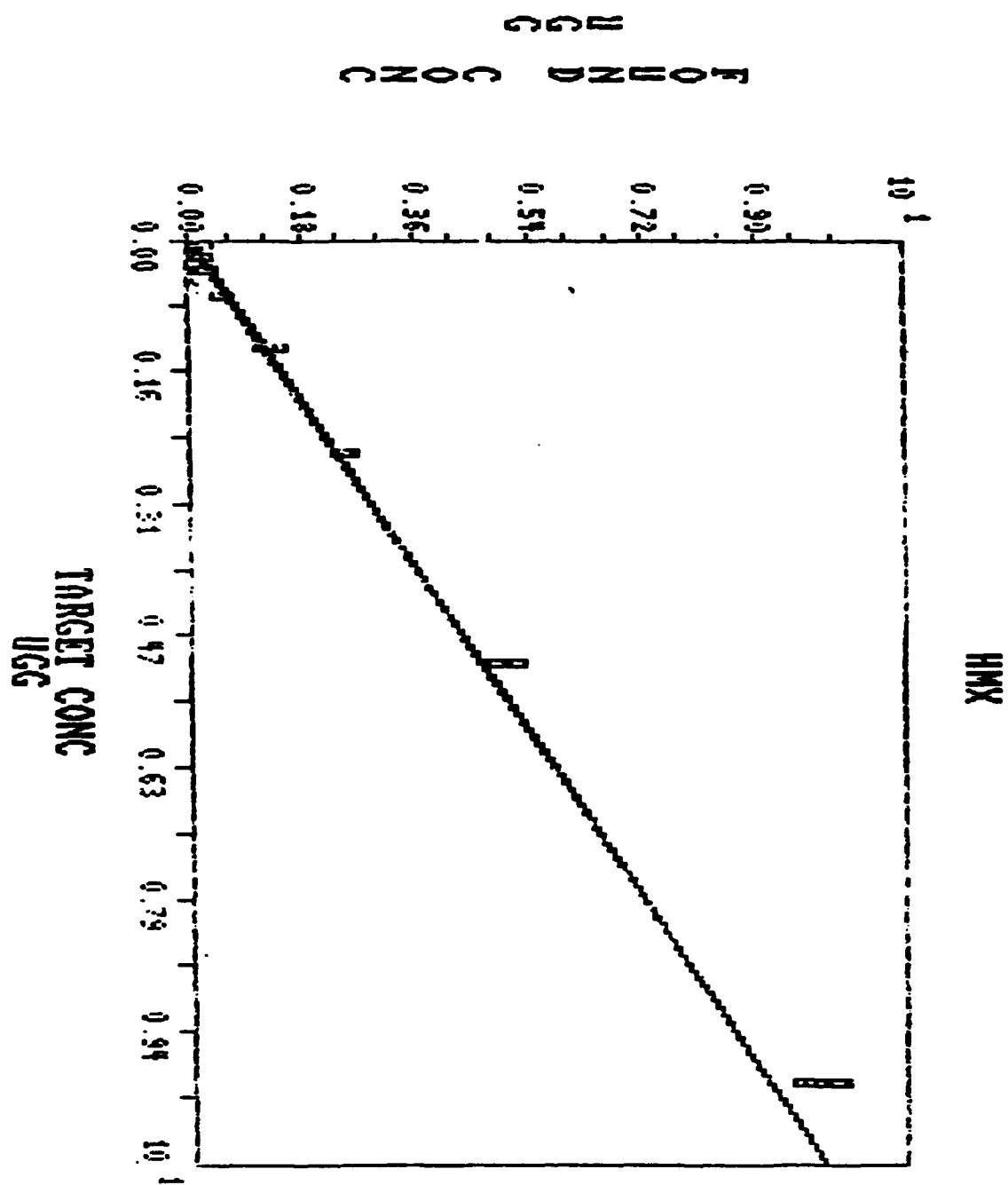


FIGURE F 26a

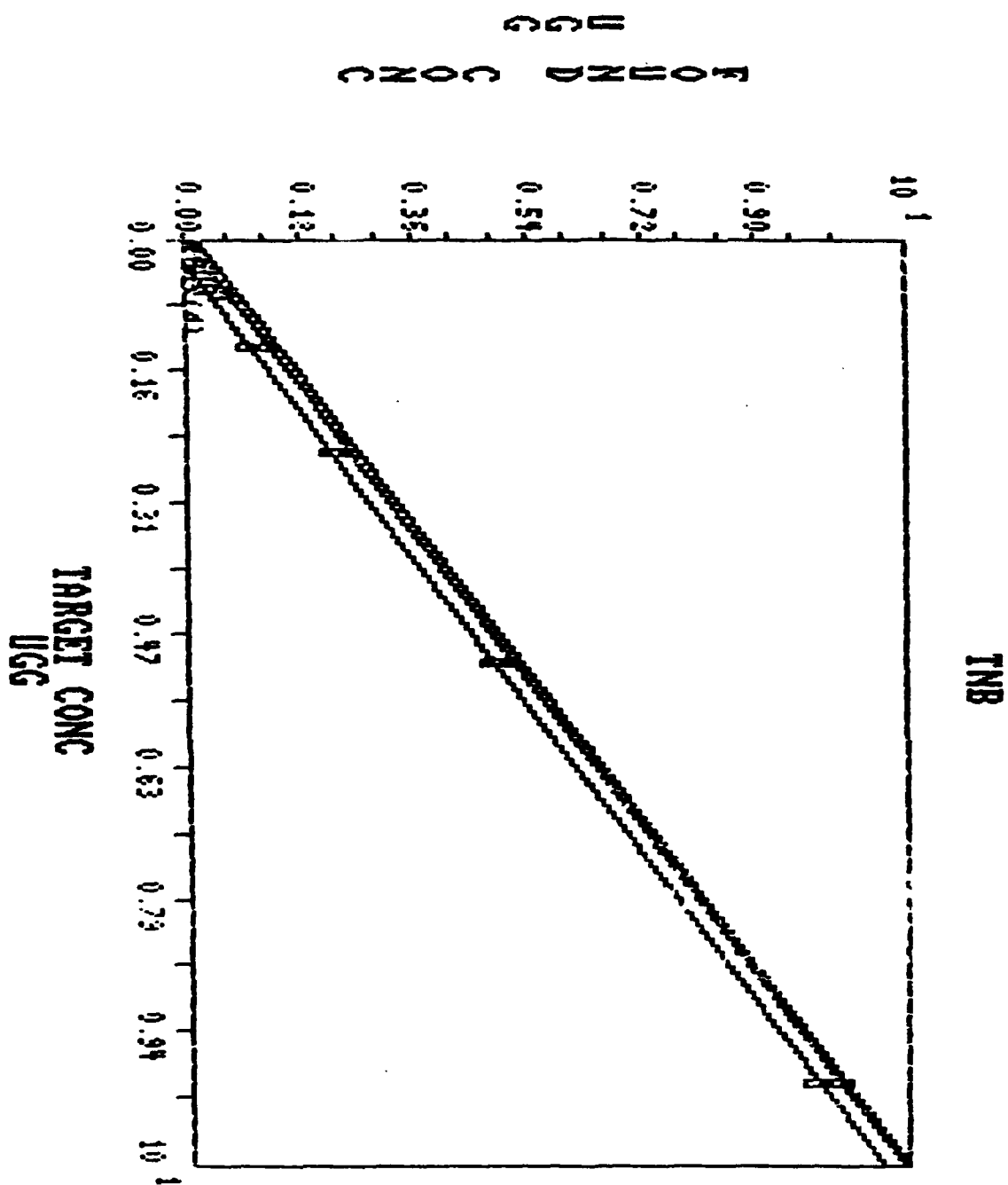


FIGURE F 26b

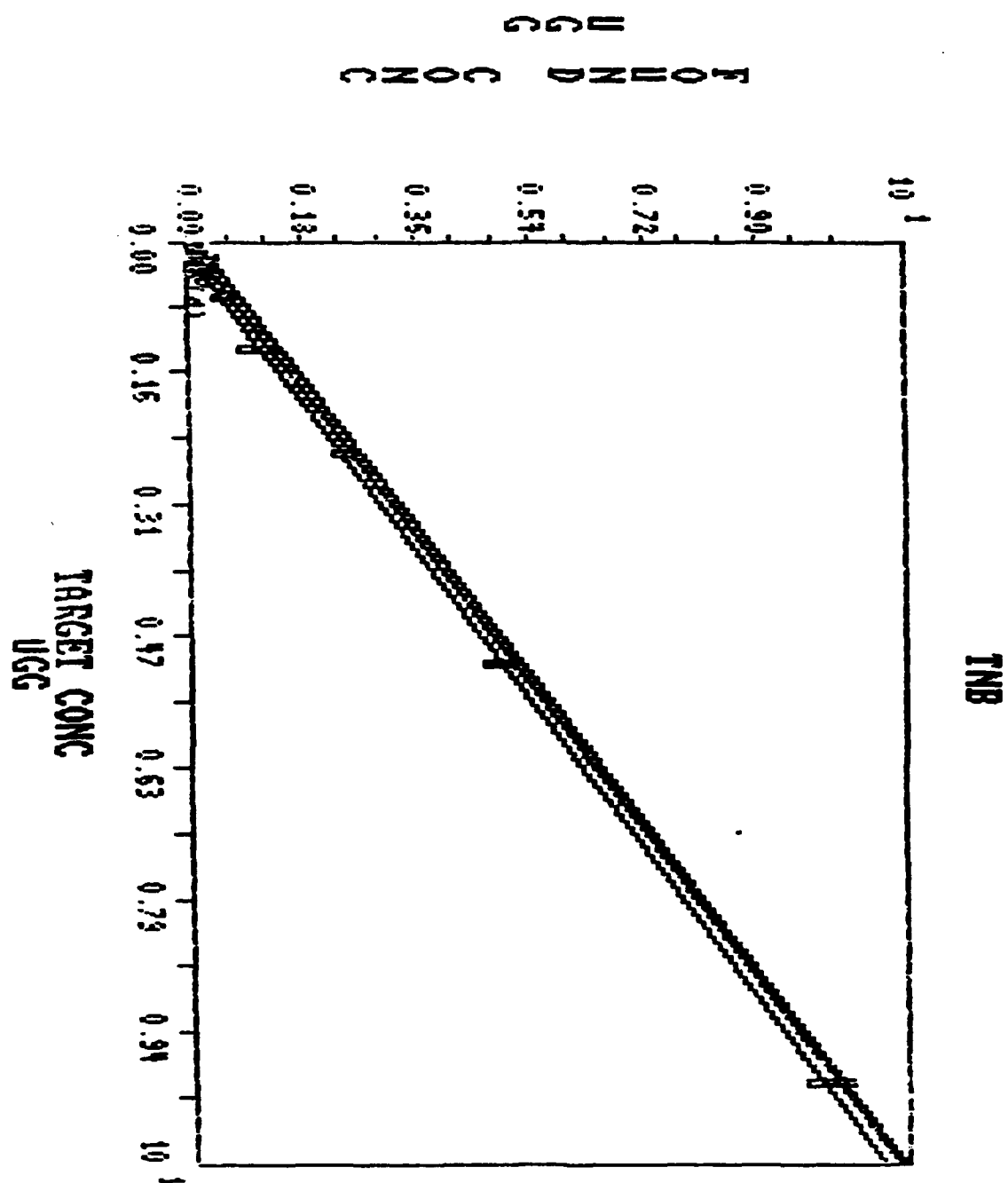


FIGURE F 27a

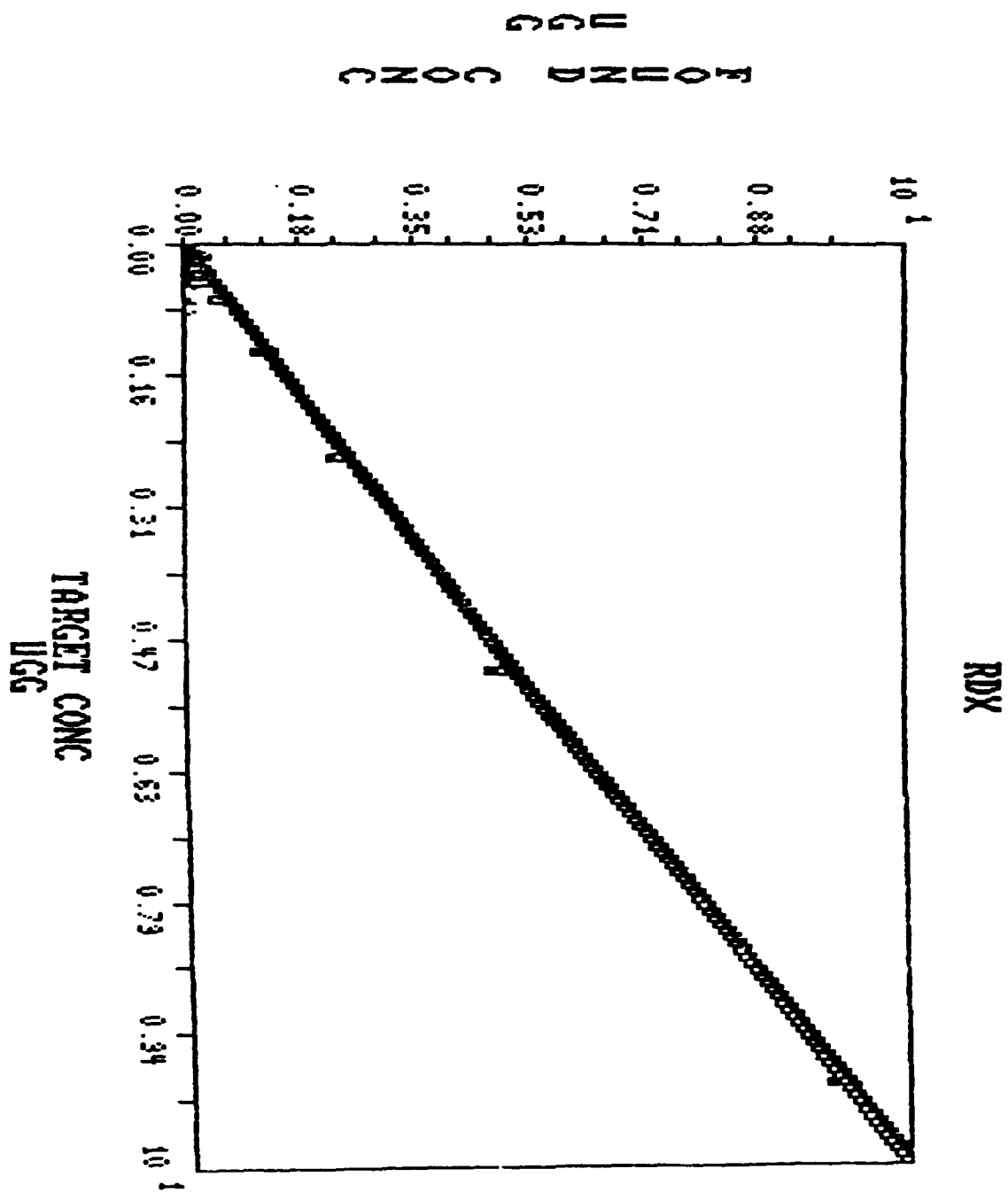


FIGURE F 27b

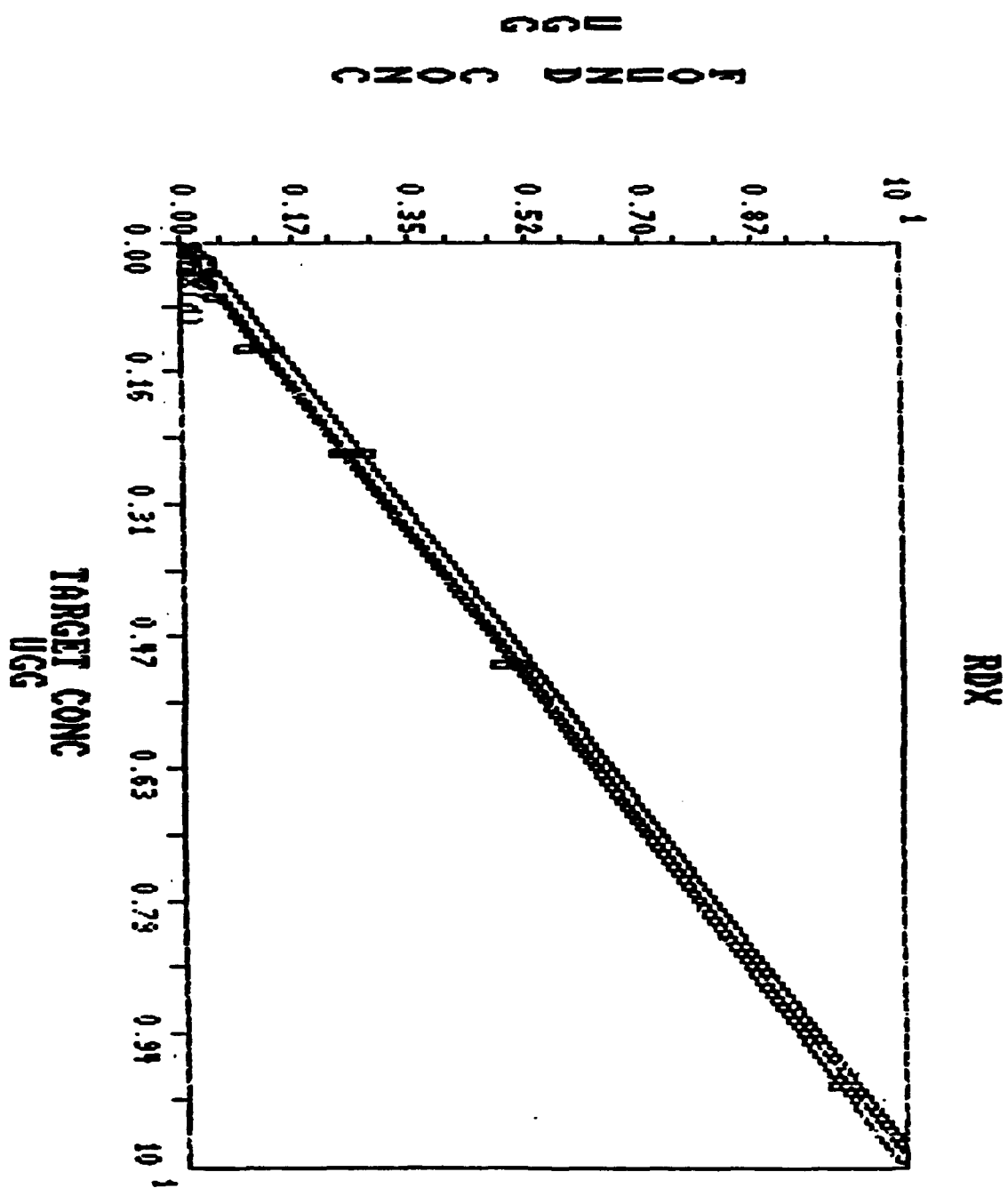


FIGURE F 28a

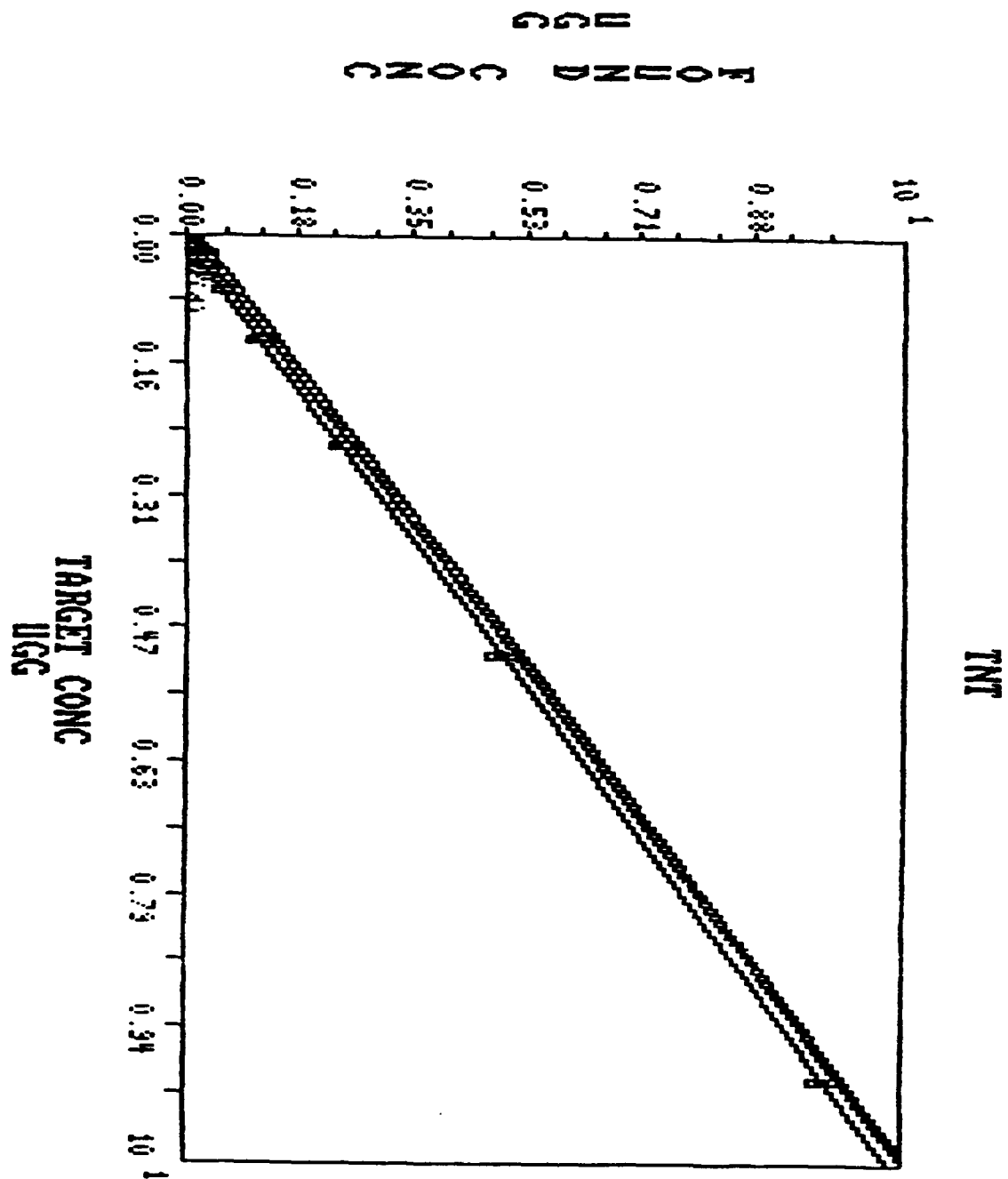


FIGURE F 28b

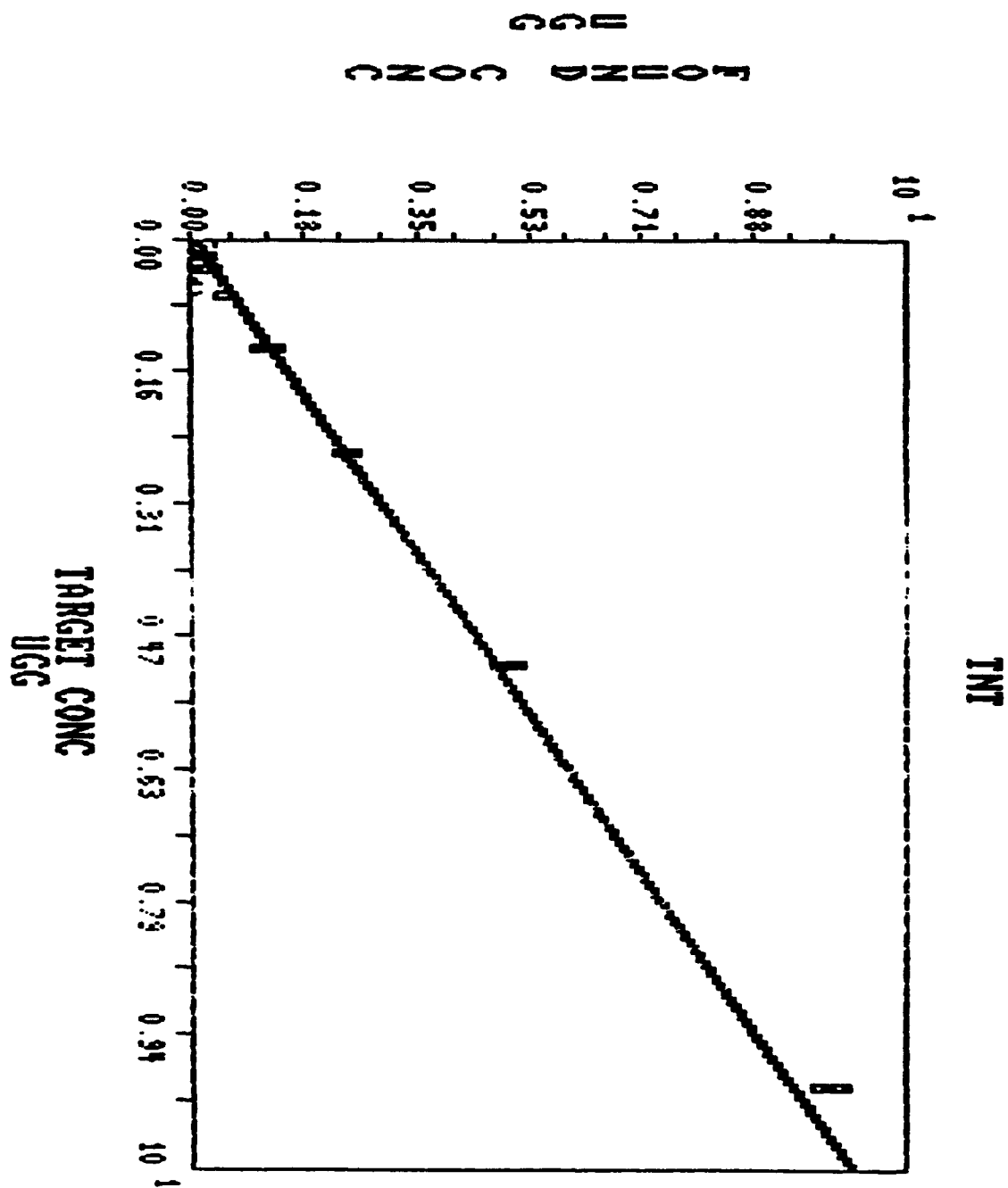


FIGURE F 29a

2,4

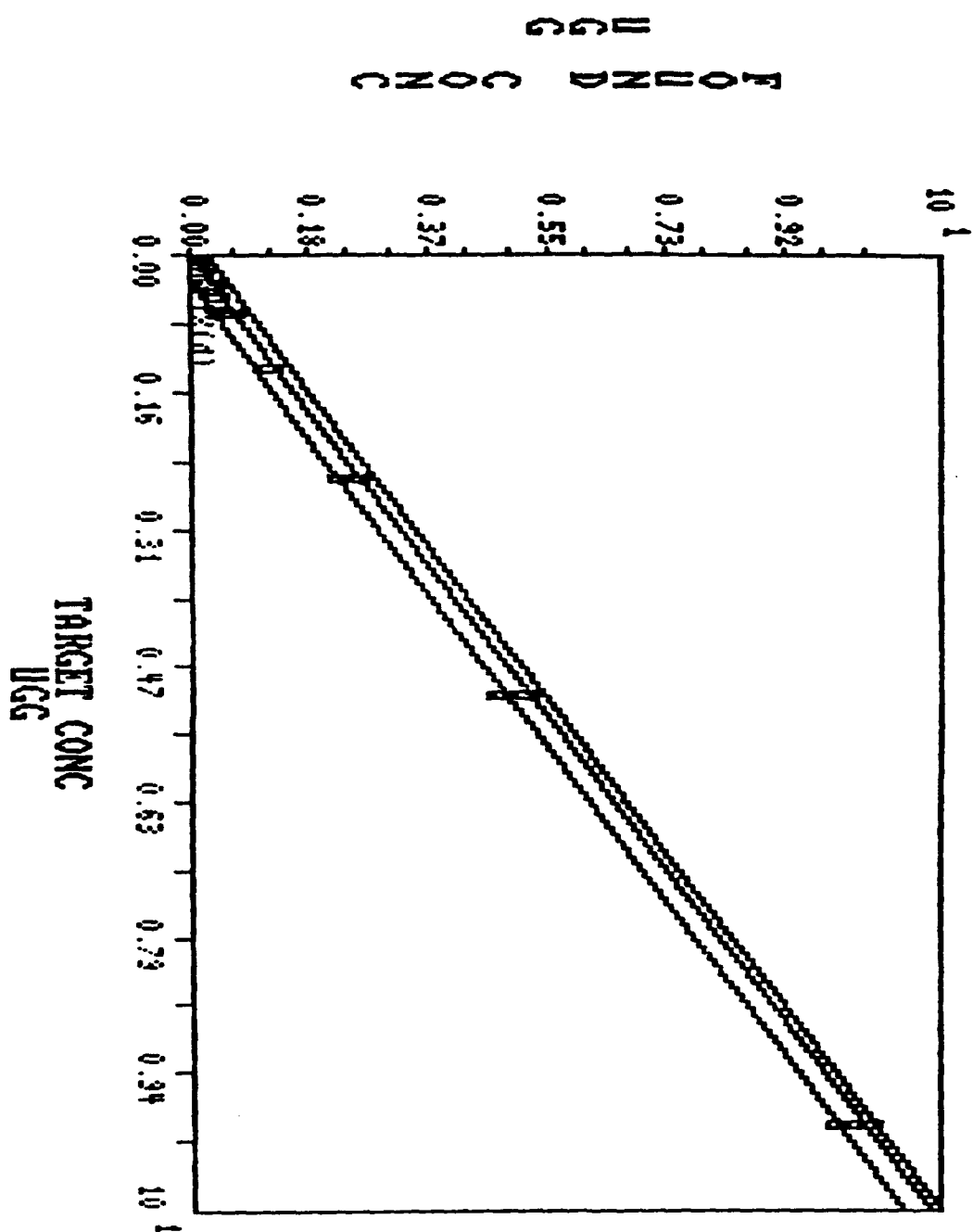


FIGURE F 29b

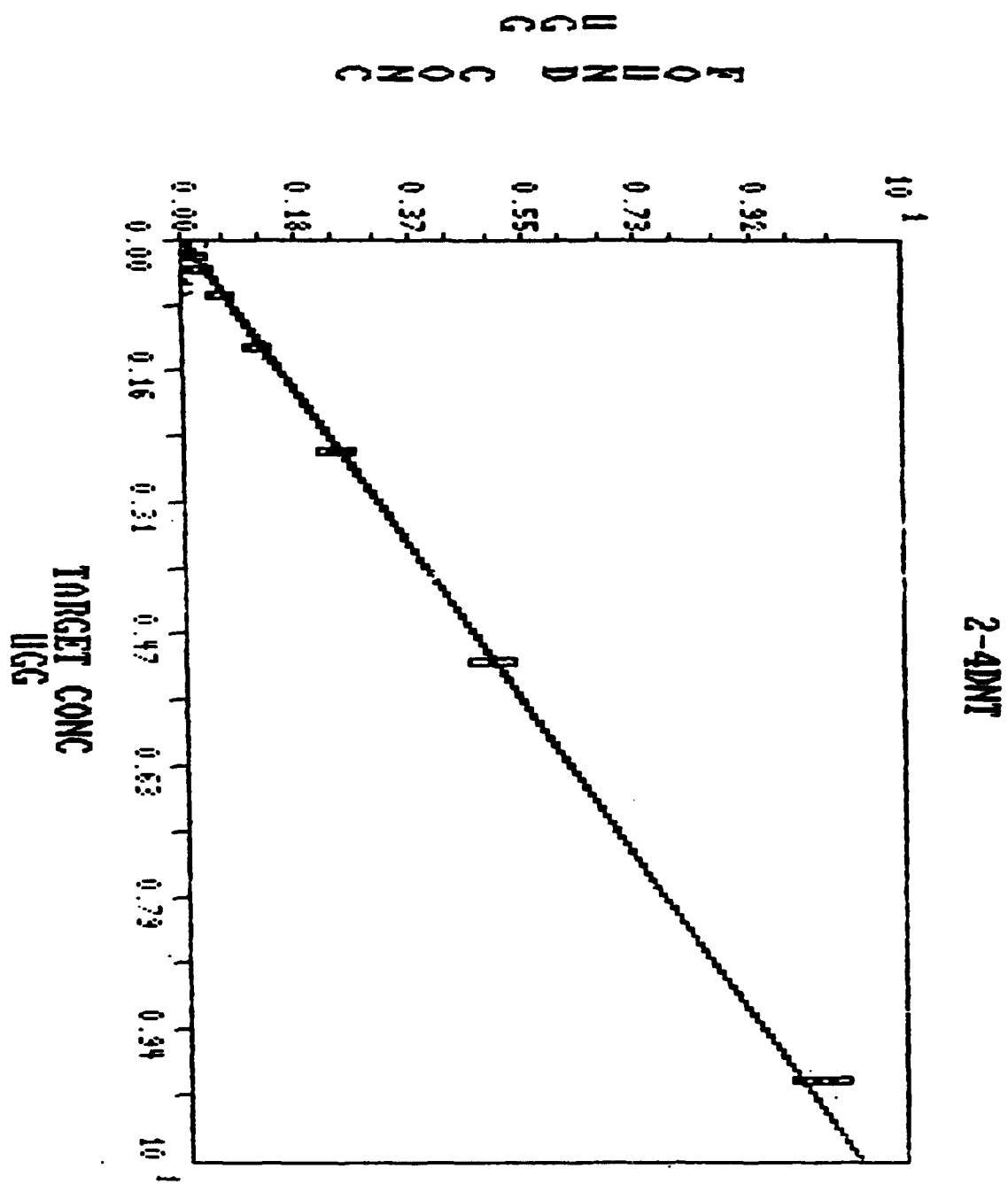


FIGURE F 30a

2,6

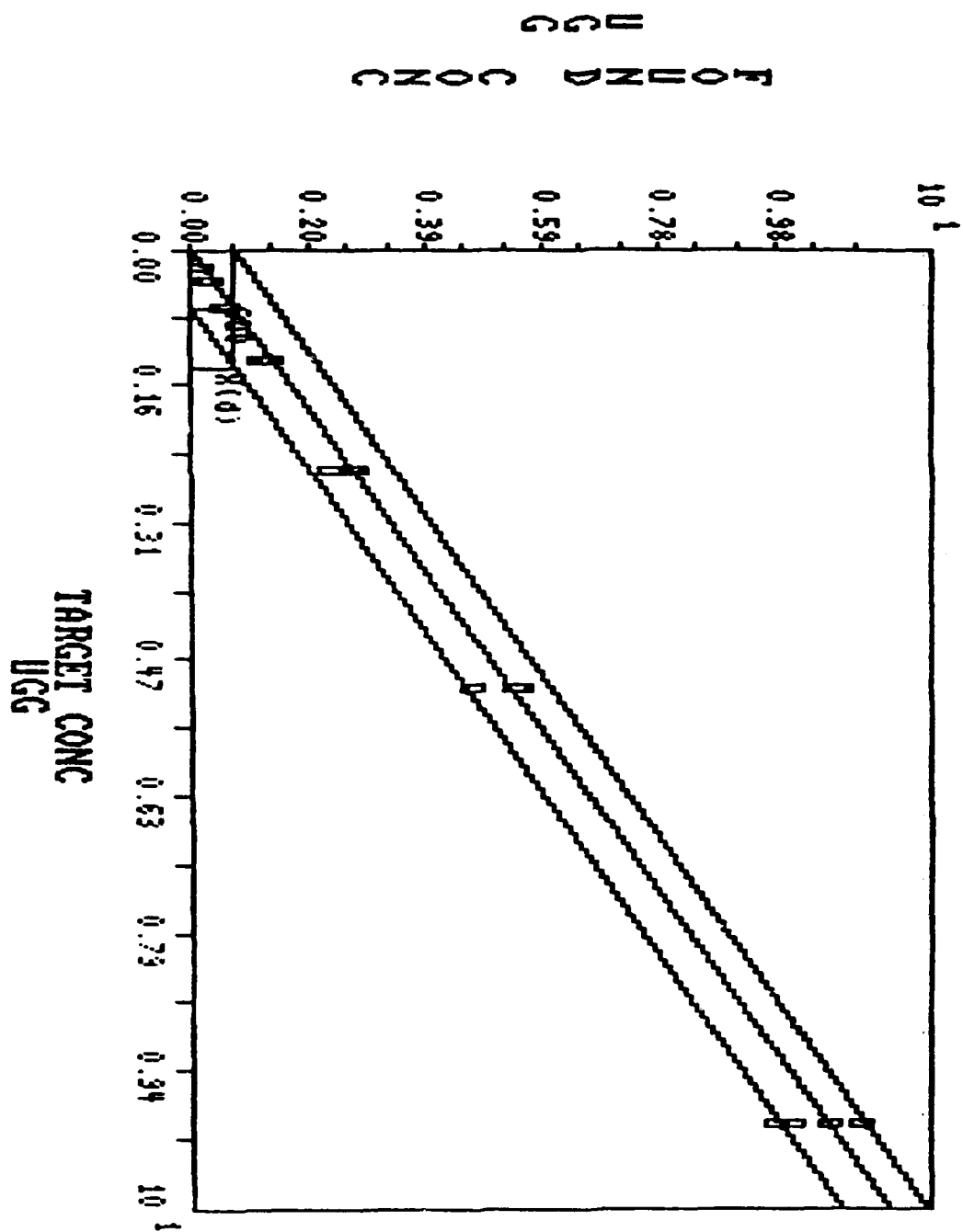


FIGURE F 30b

2-6DNT

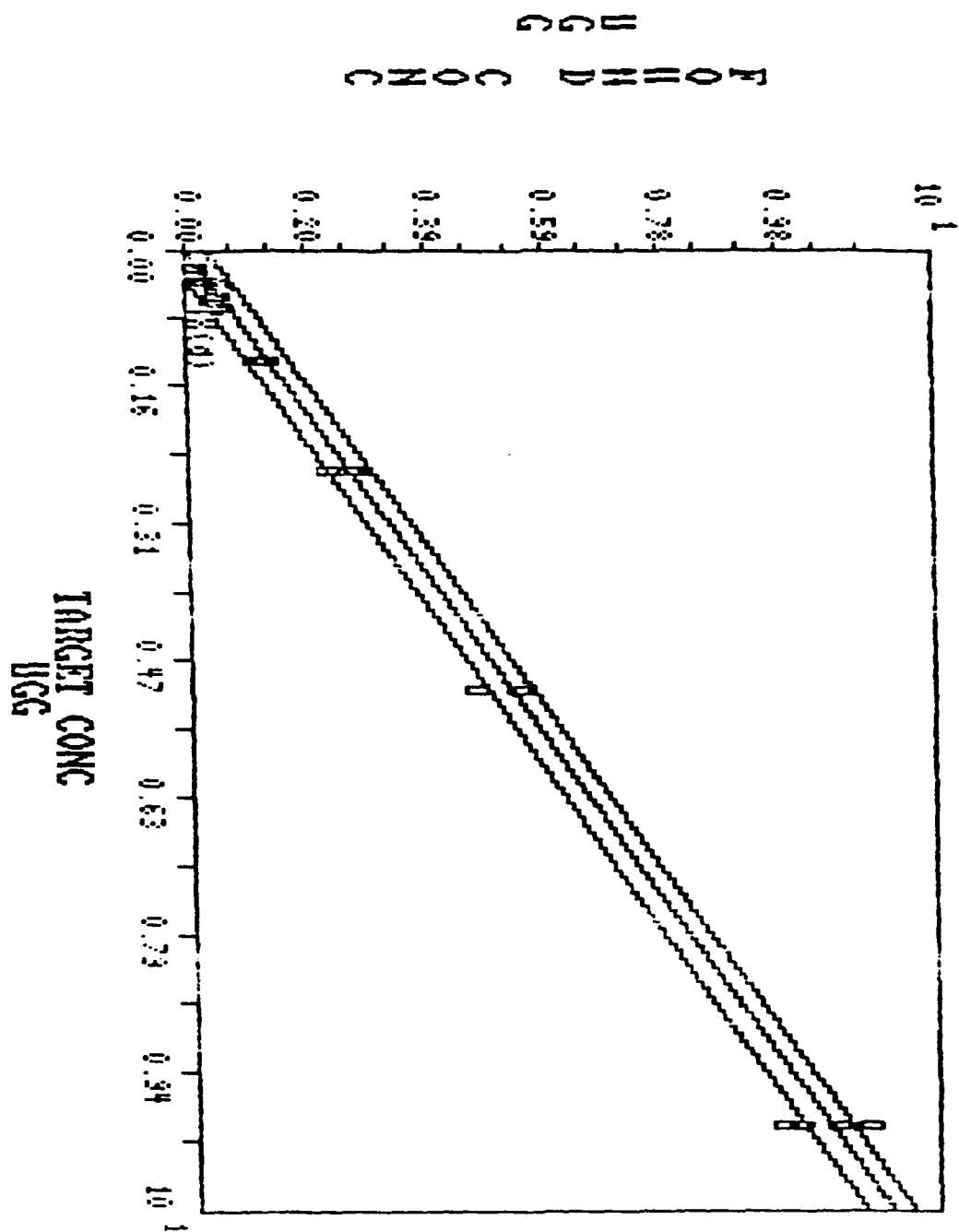


FIGURE F 31a

2AM

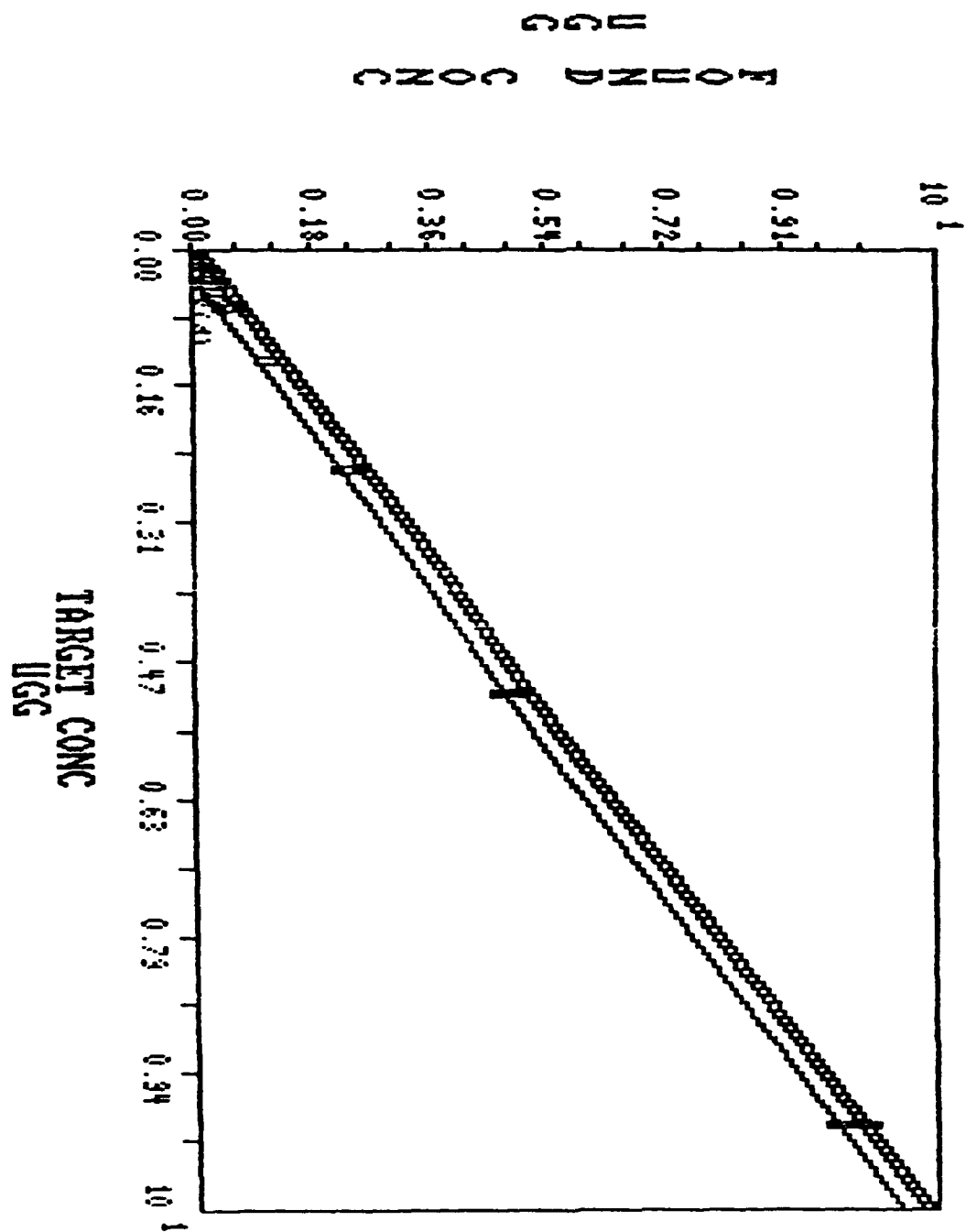


FIGURE F 31b

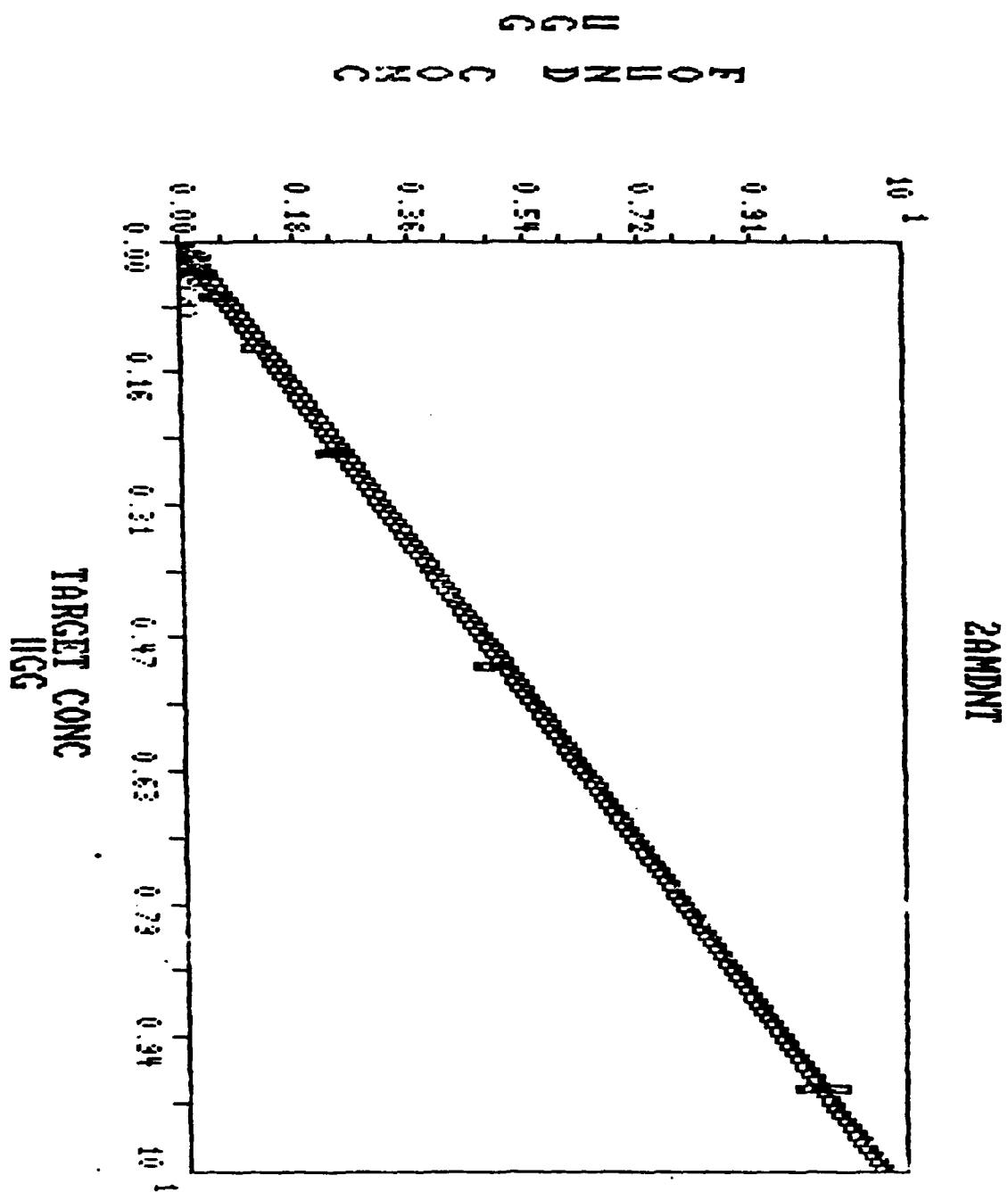


FIGURE F 32a

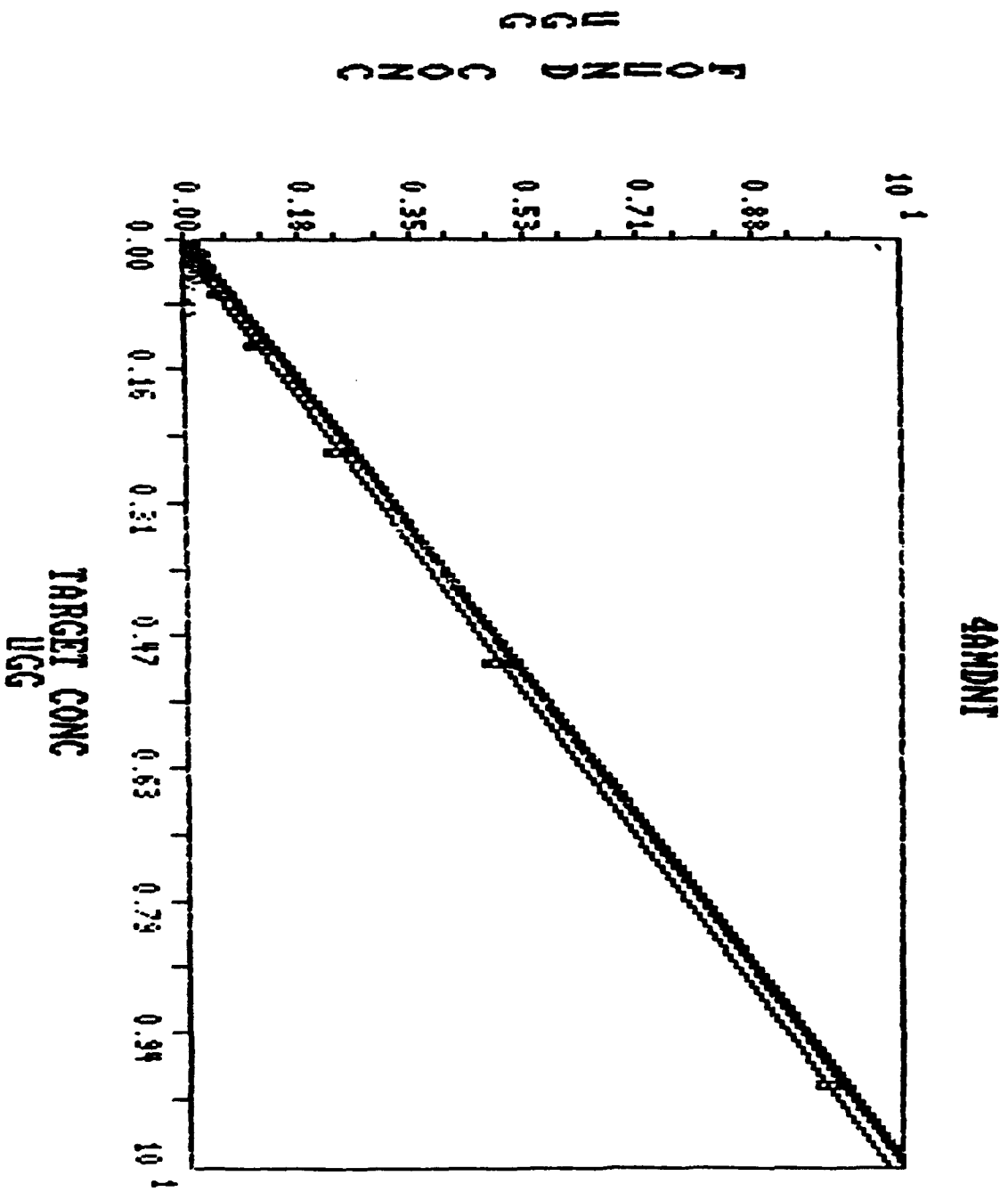


FIGURE F. 32b

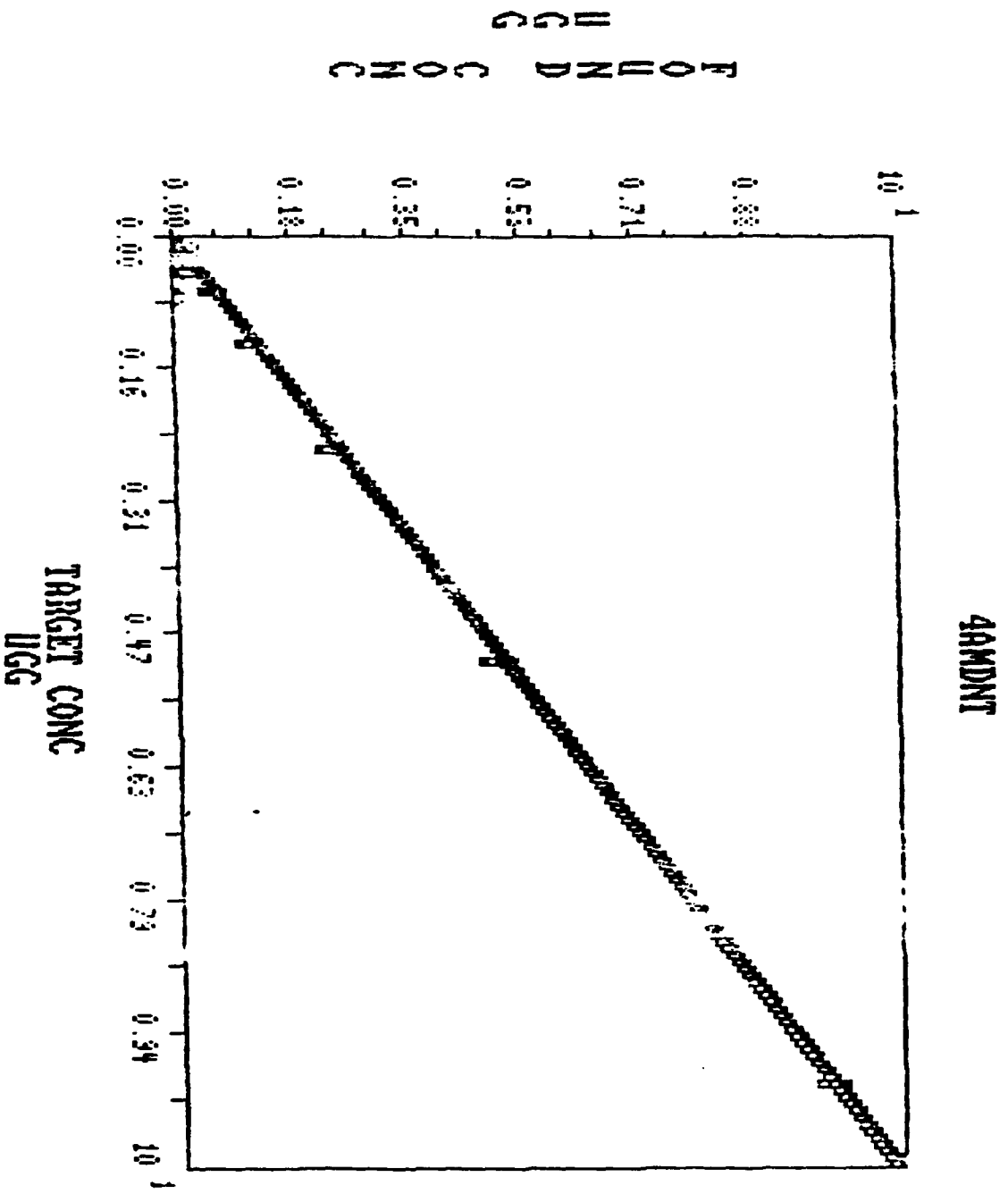


TABLE F18
CRITERION OF DETECTION WATER AND SOLVENT (mg/L)

| <u>COMPOUND</u> | <u>CD-R</u> | <u>CD-M</u> |
|-----------------|-------------|-------------|
| HMX | 0.14 | 0.14 |
| TNB | 0.13 | 0.15 |
| RDX | 0.17 | 0.07 |
| DNB | 0.15 | 0.15 |
| TNT | 0.09 | 0.09 |
| 2,4 DNT | 0.18 | 0.17 |
| 2,6 DNT | 0.35 | 0.37 |
| 2-AM | 0.14 | 0.14 |
| 4-AM | 0.10 | 0.12 |

CD-R=Detection for Radford; CD-M Detection for Milan

APPENDIX C

METAL ANALYSES FOR RAAP

Concentrations of selected metals were determined for soil from RAAP site. Samples from uncontaminated, contaminated, and contaminated/fortified soils were extracted to determine total extractable Cd, Cr, Cu, Pb, and Zn levels. Duplicate 4-g air-dried samples were heated with 20 mL of 1.0 M HNO₃ for 3 h, filtered by gravity, and diluted to a 50-mL volume with ultrapure water (reverse osmosis followed by double deionization). All extracts were analyzed for metals by atomic absorption spectrophotometry (Perkin Elmer Model 3030 AA Spectrometer). Corresponding standard solutions, and blank, duplicate and split samples were also analyzed to assure quality control. Mean values of metal levels are presented in table C-1.

Table C-1. Concentrations of selected metals from Radford Army Ammunition Plant (RAAP) soils.

| | Cd | Cr | Cu | Pb | Zn |
|-------------------------------|---------------------------------|-------------|-----------|------------|----------|
| | ----- mg kg ⁻¹ ----- | | | | |
| <u>Uncontaminated</u> | | | | | |
| | 0.54 ± 0.02 | 6.7 ± 0.1 | 6.0 ± 0.2 | 12.9 ± 0.1 | 67 ± 0.5 |
| <u>Contaminated</u> | | | | | |
| | 1.1 ± 0.05 | 10.9 ± 0.2 | 30 ± 3.9 | 183 ± 22 | 303 ± 44 |
| <u>Contaminated Fortified</u> | | | | | |
| | 0.95 ± 0.04 | 12.9 ± 0.04 | 23 ± 1.3 | 137 ± 34 | 294 ± 12 |

BLANK

APPENDIX D
RADFORD ARMY AMMUNITION PLANT
MUNITION RESIDUE DATA FROM SOIL AND LEACHATE SAMPLES

The amount of munition residue in each leachate was calculated by multiplication of the sample volume by the concentration. The amount of residue in each soil section was calculated by multiplication of the concentration of munition residue in the soil by the soil weight.

When a value of less than the criteria of detection (trace concentration) appears in tables of concentration, an "*" was entered in the corresponding amount table (concentration x leachate volume or concentration x soil weight). Zero values in the amount tables corresponded to a "none detected" (0) level in the concentration tables.

TABLE D-1.

Leachate volumes (mL) from Radford Army Ammunition Plant
(RAAP) soil columns.

| DAY # | 14 | 28 | 42 | 56 | 70 | 84 | 98 |
|-----------------|--------|--------|--------|--------|--------|--------|--------|
| POS# | AUG 3 | AUG 17 | SEP 1 | SEP 15 | SEP 29 | OCT 13 | OCT 27 |
| ----- mL ----- | | | | | | | |
| 1 | 980 | 830 | 770 | 880 | 800 | 710 | - |
| 2 | 808 | 810 | 870 | 900 | 810 | 710 | - |
| 7 | 770 | 800 | 800 | 800 | 790 | 660 | - |
| 8 | 868 | 870 | 770 | 950 | 820 | 720 | - |
| 3 | 788 | 810 | 780 | 840 | 820 | 700 | 780 |
| 4 | 947 | 810 | 820 | 930 | 830 | 710 | 790 |
| 9 | 750 | 760 | 890 | 900 | 840 | 700 | 760 |
| 10 | 998 | 850 | 820 | 890 | 820 | 710 | 780 |
| 5 | 859 | 850 | 790 | 890 | 840 | 690 | 780 |
| 6 | 874 | 900 | 820 | 850 | 840 | 690 | 790 |
| 11 | 888 | 810 | 860 | 870 | 740 | 695 | 790 |
| 12 | 930 | 910 | 490 | 970 | 899 | 800 | 700 |
| AVG. | 871.67 | 834.17 | 790.00 | 889.17 | 820.75 | 707.92 | 771.25 |
| STD. DEV. | 78.22 | 41.72 | 97.89 | 45.18 | 35.75 | 31.45 | 28.48 |
| %REL. STD. DEV. | 8.97 | 5.00 | 12.39 | 5.08 | 4.36 | 4.44 | 8.97 |
| | | | | | | | |
| DAY # | 112 | 127 | 144 | 155 | 168 | 183 | 196 |
| POS # | NOV 9 | NOV 24 | DEC 11 | DEC 22 | JAN 4 | JAN 19 | FEB 1 |
| ----- mL ----- | | | | | | | |
| 1 | - | - | - | - | - | - | - |
| 2 | - | - | - | - | - | - | - |
| 7 | - | - | - | - | - | - | - |
| 8 | - | - | - | - | - | - | - |
| 3 | 730 | 730 | 850 | 630 | 560 | 695 | - |
| 4 | 720 | 710 | 872 | 660 | 530 | 730 | - |
| 9 | 700 | 740 | 860 | 660 | 520 | 730 | - |
| 10 | 690 | 780 | 850 | 650 | 550 | 750 | - |
| 5 | 680 | 440 | 768 | 600 | 535 | 720 | 780 |
| 6 | 670 | 730 | 780 | 620 | 540 | 510 | 810 |
| 11 | 660 | 720 | 775 | 620 | 530 | 710 | 800 |
| 12 | 825 | 500 | 930 | 770 | 330 | 970 | 760 |
| AVG. | 709.38 | 668.75 | 835.63 | 651.25 | 511.88 | 726.88 | 787.50 |
| STD. DEV. | 49.02 | 117.31 | 53.13 | 49.10 | 69.73 | 116.21 | 19.20 |
| %REL. STD. DEV. | 5.00 | 12.39 | 5.08 | 4.36 | 4.44 | 3.70 | 2.44 |

TABLE D-1. Continued...

| DAY # | 210 | 225 | 239 | 253 | 267 | 274 |
|-----------------|----------------|--------|--------|--------|--------|--------|
| POS # | FEB 15 | MAR 2 | MAR 16 | MAR 30 | APR 13 | APR 20 |
| | ----- mL ----- | | | | | |
| 1 | - | - | - | - | - | - |
| 2 | - | - | - | - | - | - |
| 7 | - | - | - | - | - | - |
| 8 | - | - | - | - | - | - |
| 3 | - | - | - | - | - | - |
| 4 | - | - | - | - | - | - |
| 9 | - | - | - | - | - | - |
| 10 | - | - | - | - | - | - |
| 5 | 750 | 680 | 710 | 740 | 725 | 340 |
| 6 | 770 | 700 | 730 | 760 | 590 | 300 |
| 11 | 800 | 680 | 700 | 860 | 740 | 305 |
| 12 | 805 | 670 | 450 | 1140 | 720 | 295 |
| AVG. | 781.25 | 682.50 | 647.50 | 875.00 | 693.75 | 310.00 |
| STD. DEV. | 22.46 | 10.90 | 114.54 | 159.61 | 60.35 | 17.68 |
| %REL. STD. DEV. | 2.88 | 1.60 | 17.69 | 18.24 | 8.70 | 5.70 |

TABLE D-2. Concentrations (mg/L) of munition residues in aqueous leachates collected from RAAP soil columns.

No detectable concentrations of TNB, TNT, 2,4-DNT, 2,6-DNT, 2-amino-DNT, and 4-amino-DNT were found in any leachates collected from RAAP soil columns.

TABLE D-3. Amounts (ug) of munition residues in aqueous leachates collected from RAAP soil columns.

No detectable amounts of TNB, TNT, 2,4-DNT, 2,6-DNT, 2-amino-DNT, and 4-amino-DNT were found in any leachates collected from RAAP soil columns.

TABLE D-4.1. Concentrations (mg/kg) of munition residues in soil sections (triplicates) from RAAP soil columns, after 0 months of leaching (time zero).

| SAMPLE ID | TNB | TNT | 2,4-DNT | 2,6-DNT | 2-AM-DNT | 4-AM-DNT |
|-----------|-----|-----|---------|---------|----------|----------|
|-----------|-----|-----|---------|---------|----------|----------|

Depth (inches; 2.54-cm sections)

COLUMN #s 1,2,3,5,6,8,9,10,11 (Treatment columns)

| | | mg/kg | | | | |
|---|-----------------|-------|-------|-------|------|---|
| | | ----- | | | | |
| 1 | AVG. | 103.8 | 61.7 | 115.8 | 36.6 | 0 |
| | STD. DEV. | 1 | 7.5 | 0.2 | 0.8 | 0 |
| | %REL. STD. DEV. | 0.96 | 12.16 | 0.17 | 2.18 | 0 |

Below this depth: no detectable concentrations of munition residues.

COLUMN #s 4,7,12 (Control columns)

| | | | | | | |
|---|-----------------|---|---|---|---|---|
| 1 | AVG. | 0 | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | 0 |

At all depths: no detectable concentrations of munition residues.

TABLE D-4.2. Concentrations (mg/kg) of munition residues in soil sections (triplicates) from RAAP soil columns, after 3 months of leaching.

| SAMPLE ID | | TNB | TNT | 2,4-DNT | 2,6-DNT | 2-AM-DNT | 4-AM-DNT |
|----------------------------------|--|-----|-----|---------|---------|----------|----------|
| Depth (inches; 2.54-cm sections) | | | | | | | |

| COLUMN #1 | | mg/kg | | | | | |
|-----------|-----------------|-------|------|------|------|-------|---|
| 1 | AVG. | <2.4 | 6.61 | <5.7 | <5.2 | <15.4 | 0 |
| | STD. DEV. | - | 1.18 | - | - | - | 0 |
| | %REL. STD. DEV. | - | 17.9 | - | - | - | 0 |
| | AVG. | <2.4 | <6.1 | <5.7 | 0 | <15.4 | 0 |
| | STD. DEV. | - | - | - | 0 | - | 0 |
| | %REL. STD. DEV. | - | - | - | 0 | - | 0 |
| | AVG. | 0 | 0 | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |

Below this depth: no detectable concentrations of munition residues.

| | | | | | | | |
|-----------|-----------------|------|-------|------|---|-------|-------|
| COLUMN #2 | | | | | | | |
| 1 | AVG. | <2.4 | 7.21 | <5.7 | 0 | 0 | 0 |
| | STD. DEV. | - | 0.77 | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | - | 10.71 | - | 0 | 0 | 0 |
| 2 | AVG. | <2.4 | <6.1 | <5.7 | 0 | <15.4 | <14.6 |
| | STD. DEV. | - | - | - | 0 | - | - |
| | %REL. STD. DEV. | - | - | - | 0 | - | - |
| 3 | AVG. | 0 | 0 | <5.7 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | - | 0 | 0 | 0 |

Below this depth: no detectable concentrations of munition residues.

| | | | | | | | |
|---------------------|-----------------|---|---|---|---|---|---|
| COLUMN #7 (Control) | | | | | | | |
| 1 | AVG. | 0 | 0 | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |

At all depths: no detectable concentrations of munition residues.

TABLE D-4.2. Continued...

| SAMPLE ID | | TNB | TNT | 2,4-DNT | 2,6-DNT | 2-AM-DNT | 4-AM-DNT |
|----------------------------------|-----------------|-------|-------|---------|---------|----------|----------|
| Depth (inches; 2.54-cm sections) | | | | | | | |
| COLUMN #8 | | mg/kg | | | | | |
| 1 | AVG. | <2.4 | 8.5 | <5.7 | 0 | 0 | 0 |
| | STD. DEV. | - | 1 | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | - | 11.77 | - | 0 | 0 | 0 |
| 2 | AVG. | <2.4 | <6.1 | <5.7 | 0 | <15.4 | 0 |
| | STD. DEV. | - | - | - | 0 | - | 0 |
| | %REL. STD. DEV. | - | - | - | 0 | - | 0 |
| 3 | AVG. | 0 | 0 | 0 | 0 | <15.4 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | - | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | - | 0 |

Below this depth: no detectable concentrations of munition residues.

TABLE D-4.3. Concentrations (mg/kg) of munition residues in soil sections (triplicates) from RAAP soil columns, after 6 months of leaching

| SAMPLE ID | | TNB | TNT | 2,4-DNT | 2,6-DNT | 2-AM-DNT | 4-AM-DNT |
|----------------------------------|-----------------|-------------------|------|---------|---------|----------|----------|
| Depth (inches; 2.54-cm sections) | | | | | | | |
| COLUMN #3 | | ----- mg/kg ----- | | | | | |
| 1 | AVG. | <2.4 | <6.1 | <5.7 | 0 | 0 | 0 |
| | STD. DEV. | - | - | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | - | - | - | 0 | 0 | 0 |
| 2 | AVG. | 0 | <6.1 | <5.7 | 0 | 0 | 0 |
| | STD. DEV. | 0 | - | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | - | - | 0 | 0 | 0 |
| 3 | AVG. | 0 | 0 | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |

Below this depth: no detectable concentrations of munition residues.

COLUMN #4 (Control)

| | | | | | | | |
|---|-----------------|---|---|---|---|---|---|
| 1 | AVG. | 0 | 0 | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |

At all depths: no detectable concentrations of munition residues.

COLUMN #9

| | | | | | | | |
|---|-----------------|------|------|------|---|---|---|
| 1 | AVG. | <2.4 | <6.1 | <5.7 | 0 | 0 | 0 |
| | STD. DEV. | - | - | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | - | - | - | 0 | 0 | 0 |
| 2 | AVG. | <2.4 | <6.1 | <5.7 | 0 | 0 | 0 |
| | STD. DEV. | - | - | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | - | - | - | 0 | 0 | 0 |
| 3 | AVG. | 0 | 0 | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |

Below this depth: no detectable concentrations of munition residues.

TABLE D-4.3. Continued...

| SAMPLE ID | | TNB | TNT | 2,4-DNT | 2,6-DNT | 2-AM-DNT | 4-AM-DNT |
|----------------------------------|-----------------|-------|------|---------|---------|----------|----------|
| Depth (inches; 2.54-cm sections) | | | | | | | |
| COLUMN #10 | | mg/kg | | | | | |
| 1 | AVG. | <2.4 | 7.84 | <5.7 | 0 | 0 | 0 |
| | STD. DEV. | - | 1.97 | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | - | 25.1 | - | 0 | 0 | 0 |
| 2 | AVG. | 0 | <6.1 | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | - | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | - | 0 | 0 | 0 | 0 |
| 3 | AVG. | 0 | 0 | <5.7 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | - | 0 | 0 | 0 |

Below this depth: no detectable concentrations of munition residues.

TABLE D-4.4. Concentrations (mg/kg) of munition residues in soil sections
(triplicates) from RAAP soil columns, after 9 months of leaching.

| SAMPLE ID | | TNB | TNT | 2,4-DNT | 2,6-DNT | 2-AM-DNT | 4-AM-DNT |
|----------------------------------|--|-----|-----|---------|---------|----------|----------|
| Depth (inches; 2.54-cm sections) | | | | | | | |

| COLUMN #5 | | mg/kg | | | | | |
|-----------|-----------------|-------|------|------|---|-------|---|
| 1 | AVG. | <2.4 | <6.1 | <5.7 | 0 | 0 | 0 |
| | STD. DEV. | - | - | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | - | - | - | 0 | 0 | 0 |
| 2 | AVG. | <2.4 | <6.1 | <5.7 | 0 | <15.4 | 0 |
| | STD. DEV. | - | - | - | 0 | - | 0 |
| | %REL. STD. DEV. | - | - | - | 0 | - | 0 |
| 3 | AVG. | 0 | 0 | 0 | 0 | <15.4 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | - | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | - | 0 |

Below this depth: no detectable concentrations of munition residues.

COLUMN #6

| | | | | | | | |
|---|-----------------|------|-------|------|------|-------|---|
| 1 | AVG. | <2.4 | 10.44 | <5.7 | <5.2 | <15.4 | 0 |
| | STD. DEV. | - | 4.33 | - | - | - | 0 |
| | %REL. STD. DEV. | - | 41.53 | - | - | - | 0 |
| 2 | AVG. | <2.4 | <6.1 | <5.7 | <5.2 | <15.4 | 0 |
| | STD. DEV. | - | - | - | - | - | 0 |
| | %REL. STD. DEV. | - | - | - | - | - | 0 |
| 3 | AVG. | 0 | 0 | 0 | 0 | <15.4 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | - | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | - | 0 |

Below this depth: no detectable concentrations of munition residues.

TABLE D-4.4. Continued...

| SAMPLE ID | | TNB | TNT | 2,4-DNT | 2,6-DNT | 2-AM-DNT | 4-AM-DNT |
|----------------------------------|--|-----|-----|---------|---------|----------|----------|
| Depth (inches; 2.54-cm sections) | | | | | | | |

| COLUMN #11 | | mg/kg | | | | | |
|------------|-----------------|-------|------|------|------|-------|---|
| 1 | AVG. | <2.4 | <6.1 | <5.7 | <5.2 | <15.4 | 0 |
| | STD. DEV. | - | - | - | - | - | 0 |
| | %REL. STD. DEV. | - | - | - | - | - | 0 |
| 2 | AVG. | <2.4 | <6.1 | <5.7 | 0 | <15.4 | 0 |
| | STD. DEV. | - | - | - | 0 | - | 0 |
| | %REL. STD. DEV. | - | - | - | 0 | - | 0 |
| 3 | AVG. | 0 | 0 | <5.7 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | - | 0 | 0 | 0 |

Below this depth: no detectable concentrations of munition residues.

COLUMN #12 (Control)

| | | | | | | | |
|---|-----------------|---|---|---|---|---|---|
| 1 | AVG. | 0 | 0 | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |

At all depths: no detectable concentrations of munition residues.

TABLE D-4.5. Amounts (ug) of munition residues in each soil-core section (triplicates) from RAAP soil columns, after 0 months of leaching (time zero).

| SAMPLE ID | TNB | TNT | 2,4-DNT | 2,6-DNT | 2-AM-DNT | 4-AM-DNT |
|----------------------------------|-----|-----|---------|---------|----------|----------|
| Depth (inches; 2.54-cm sections) | | | | | | |

COLUMN #s 1,2,3,5,6,8,9,10,11 (Treatment columns)

| | | ----- ug ----- | | | ----- | | |
|---|-----------------|----------------|-------|-------|-------|---|---|
| 1 | AVG. | 25950 | 15425 | 28950 | 9150 | 0 | 0 |
| | STD. DEV. | 250 | 1875 | 50 | 200 | 0 | 0 |
| | %REL. STD. DEV. | 0.96 | 12.16 | 0.173 | 2.18 | 0 | 0 |

Below this depth: no detectable concentrations of munition residues.

COLUMN #s 4,7,12 (Control columns)

| | | | | | | | |
|---|-----------------|---|---|---|---|---|---|
| 1 | AVG. | 0 | 0 | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |

At all depths: no detectable concentrations of munition residues.

TABLE D-4.6. Amounts (ug) of munition residues in each soil-core section
(triplicates) from RAAP soil columns, after 3 months of leaching.

| SAMPLE ID | TNB | TNT | 2,4-DNT | 2,6-DNT | 2-AM-DNT | 4-AM-DNT |
|----------------------------------|-----|-----|---------|---------|----------|----------|
| Depth (inches; 2.54-cm sections) | | | | | | |

| COLUMN #1 | ----- ug ----- | | | | | |
|-----------|-----------------|---|--------|---|---|---|
| 1 | AVG. | * | 1859 | * | * | 0 |
| | STD. DEV. | - | 333.58 | - | - | 0 |
| | %REL. STD. DEV. | - | 17.9 | - | - | 0 |
| 2 | AVG. | * | * | * | 0 | 0 |
| | STD. DEV. | - | - | - | 0 | 0 |
| | %REL. STD. DEV. | - | - | - | 0 | 0 |
| 3 | AVG. | 0 | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | 0 |

Below this depth: no detectable concentrations of munition residues.

| COLUMN #2 | | | | | | | |
|-----------|-----------------|---|---------|---|---|---|---|
| 1 | AVG. | * | 2025.79 | * | 0 | 0 | 0 |
| | STD. DEV. | - | 217.1 | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | - | 10.71 | - | 0 | 0 | 0 |
| 2 | AVG. | * | * | * | 0 | * | * |
| | STD. DEV. | - | - | - | 0 | - | - |
| | %REL. STD. DEV. | - | - | - | 0 | - | - |
| 3 | AVG. | 0 | 0 | * | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | - | 0 | 0 | 0 |

Below this depth: no detectable concentrations of munition residues.

| COLUMN #7 (Control) | | | | | | | |
|---------------------|-----------------|---|---|---|---|---|---|
| 1 | AVG. | 0 | 0 | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |

At all depths: no detectable concentrations of munition residues.

* No quantifiable concentrations of munition residues.

TABLE D-4.6. Continued...

| SAMPLE ID | | TNB | TNT | 2,4-DNT | 2,6-DNT | 2-AM-DNT | 4-AM-DNT |
|----------------------------------|-----------------|-------|---------|---------|---------|----------|----------|
| Depth (inches; 2.54-cm sections) | | | | | | | |
| COLUMN #8 | | ----- | | ug | ----- | | |
| 1 | AVG. | * | 2390.44 | * | 0 | 0 | 0 |
| | STD. DEV. | - | 281.38 | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | - | 11.77 | - | | | |
| 2 | AVG. | * | * | * | 0 | * | 0 |
| | STD. DEV. | - | - | - | 0 | - | 0 |
| | %REL. STD. DEV. | - | - | - | 0 | - | 0 |
| 3 | AVG. | 0 | 0 | 0 | 0 | * | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | - | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | - | 0 |

Below this depth: no detectable concentrations of munition residues.

* No quantifiable concentrations of munition residues.

TABLE D-4.7. Amounts (ug) of munition residues in each soil-core section
(triplicates) from RAAP soil columns, after 6 months of leaching.

| SAMPLE ID | TNB | TNT | 2,4-DNT | 2,6-DNT | 2-AM-DNT | 4-AM-DNT |
|----------------------------------|-----|-----|---------|---------|----------|----------|
| Depth (inches; 2.54-cm sections) | | | | | | |

| COLUMN #3 | | ----- ug ----- | | | | |
|-----------|-----------------|----------------|---|---|---|---|
| 1 | AVG. | * | * | * | 0 | 0 |
| | STD. DEV. | - | - | - | 0 | 0 |
| | %REL. STD. DEV. | - | - | - | 0 | 0 |
| 2 | AVG. | 0 | * | * | 0 | 0 |
| | STD. DEV. | 0 | - | - | 0 | 0 |
| | %REL. STD. DEV. | 0 | - | - | 0 | 0 |
| 3 | AVG. | 0 | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | 0 |

Below this depth: no detectable concentrations of munition residues.

COLUMN #4 (Control)

| | | | | | | |
|---|-----------------|---|---|---|---|---|
| 1 | AVG. | 0 | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | 0 |

At all depths: no detectable concentrations of munition residues.

COLUMN #9

| | | | | | | |
|---|-----------------|---|---|---|---|---|
| 1 | AVG. | * | * | * | 0 | 0 |
| | STD. DEV. | - | - | - | 0 | 0 |
| | %REL. STD. DEV. | - | - | - | 0 | 0 |
| 2 | AVG. | * | * | * | 0 | 0 |
| | STD. DEV. | - | - | - | 0 | 0 |
| | %REL. STD. DEV. | - | - | - | 0 | 0 |
| 3 | AVG. | 0 | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | 0 |

Below this depth: no detectable concentrations of munition residues.

* No quantifiable concentrations of munition residues.

TABLE D-4.7. Continued...

| SAMPLE ID | | TNB | TNT | 2,4-DNT | 2,6-DNT | 2-AM-DNT | 4-AM-DNT |
|----------------------------------|-----------------|-------|---------|---------|---------|----------|----------|
| Depth (inches; 2.54-cm sections) | | | | | | | |
| COLUMN #10 | | ----- | | | ug | ----- | |
| 1 | AVG. | * | 2202.94 | * | 0 | 0 | 0 |
| | STD. DEV. | - | 552.88 | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | - | 25.1 | - | 0 | 0 | 0 |
| 2 | AVG. | 0 | * | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | - | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | - | 0 | 0 | 0 | 0 |
| 3 | AVG. | 0 | 0 | * | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | - | 0 | 0 | 0 |

Below this depth: no detectable concentrations of munition residues.

* No quantifiable concentrations of munition residues.

TABLE D-4.8. Amounts (ug) of munition residues in each soil-core section
(triplicates) from RAAP soil columns, after 9 months of leaching.

| SAMPLE ID | TNB | TNT | 2,4-DNT | 2,6-DNT | 2-AM-DNT | 4-AM-DNT |
|----------------------------------|-----|-----|---------|---------|----------|----------|
| Depth (inches; 2.54-cm sections) | | | | | | |

| COLUMN #5 | | ----- ug ----- | | | | |
|-----------|-----------------|----------------|---|---|---|---|
| 1 | AVG. | * | * | * | 0 | 0 |
| | STD. DEV. | - | - | - | 0 | 0 |
| | %REL. STD. DEV. | - | - | - | 0 | 0 |
| 2 | AVG. | * | * | * | 0 | * |
| | STD. DEV. | - | - | - | 0 | - |
| | %REL. STD. DEV. | - | - | - | 0 | - |
| 3 | AVG. | 0 | 0 | 0 | 0 | * |
| | STD. DEV. | 0 | 0 | 0 | 0 | - |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | - |

Below this depth: no detectable concentrations of munition residues.

| COLUMN #6 | | | | | | |
|-----------|-----------------|---|---------|---|---|---|
| 1 | AVG. | * | 2936.38 | * | * | * |
| | STD. DEV. | - | 1219.53 | - | - | - |
| | %REL. STD. DEV. | - | 41.53 | - | - | - |
| 2 | AVG. | * | * | * | * | * |
| | STD. DEV. | - | - | - | - | - |
| | %REL. STD. DEV. | - | - | - | - | - |
| 3 | AVG. | 0 | 0 | 0 | 0 | * |
| | STD. DEV. | 0 | 0 | 0 | 0 | - |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | - |

Below this depth: no detectable concentrations of munition residues.

* No quantifiable concentrations of munition residues.

TABLE D-4.8. Continued...

| SAMPLE ID | | TNB | TNT | 2,4-DNT | 2,6-DNT | 2-AM-DNT | 4-AM-DNT |
|----------------------------------|-----------------|-------|-----|---------|---------|----------|----------|
| Depth (inches; 2.54-cm sections) | | | | | | | |
| COLUMN #11 | | ----- | | | ug | ----- | |
| 1 | AVG. | * | * | * | * | * | 0 |
| | STD. DEV. | - | - | - | - | - | 0 |
| | %REL. STD. DEV. | - | - | - | - | - | 0 |
| 2 | AVG. | * | * | * | 0 | * | 0 |
| | STD. DEV. | - | - | - | 0 | - | 0 |
| | %REL. STD. DEV. | - | - | - | 0 | - | 0 |
| 3 | AVG. | 0 | 0 | * | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | - | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | - | 0 | 0 | 0 |

Below this depth: no detectable concentrations of munition residues.

COLUMN #12 (control)

| | | | | | | | |
|---|-----------------|---|---|---|---|---|---|
| 1 | AVG. | 0 | 0 | 0 | 0 | 0 | 0 |
| | STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |
| | %REL. STD. DEV. | 0 | 0 | 0 | 0 | 0 | 0 |

At all depths: no detectable concentrations of munition residues.